## Evaluation of the Limited Engineering Scale Testing of the PLASMOX® Technology to Treat Chemical Warfare Materiel

Prepared for:

Non-Stockpile Chemical Materiel
Program Manager

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Stone & Webster, Inc. A Shaw Group Company

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# EVALUATION OF THE LIMITED ENGINEERING SCALE TESTING OF THE **PLASMOX®** TECHNOLOGY TO TREAT CHEMICAL WARFARE MATERIEL

Prepared for

United States Army

Non-Stockpile Chemical Materiel Program Manager

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### **EXECUTIVE SUMMARY**

This report evaluates the results of the Limited Engineering Scale Tests of the Burns and Roe/MGC PLASMOX® Plasma (PLASMOX®) process. Stone & Webster conducted these tests on behalf of Non-Stockpile Chemical Materiel Program (NSCMP) (Edward F. Doyle, Alternative Systems Demonstration and Evaluation Group Leader) to assess the operability of the PLASMOX® process when treating NSCMP feeds. This included an assessment of the stability and throughput capabilities of the system when treating NSCMP liquid waste streams. The tests were conducted by Burns and Roe/MGC as part of the systemization of the PLASMOX® Research and Integration Facility 2 (RIF-2) unit during the weeks of January 8 through January 19, 2001.

The principal objective of the Limited Engineering Scale Tests was to assess the operability of the PLASMOX<sup>®</sup> plasma system to treat NSCMP neutralents. Two NSCMP MEA-based neutralent simulants were tested. The technology was evaluated based on five test objectives:

- 1) Maximum throughput for each feed.
- 2) Continuous, stable operability of the PLASMOX® System for each feed type.
- 3) Ability to process feeds such that process effluents can be disposed in a RCRA facility without the need for additional treatment.
- 4) Fate of phosphorus.
- 5) Obtain engineering data to support preliminary design.

Data and observations from tests were evaluated in accordance with the above objectives. The conclusions based on these criteria are summarized below.

- The test data show that a PLASMOX<sup>®</sup> unit equivalent in size to the RIF-2 unit could process more than 14,000 gallons of NSCMP neutralent per year, assuming the demonstrated 13 L/hr processing rate and 50% availability. The maximum continuous flow demonstrated on MEA-based neutralent simulant was equivalent to 82 gallons/day.
- The RIF-2 unit operated continuously for all but one of the test runs. The unit was stopped prematurely due to a liquid feed system disturbance during a work-up run prior to the commencement of test run data collection. Aspirating air introduced with the liquid feed upset the control of the torch electrode. During test run operations, Test Run GB-2 was aborted after 6 hours of operation because of slag deposits on the crucible rim that caused it to bind against the reactor lid. The test was scheduled for 12 hours. Both incidents were caused by non-standard operating conditions. The liquid feed system was modified to eliminate the use of aspirated air. The location of slag feed was changed after Test Run GB-2. Both of these operational changes eliminated further upsets. For the remainder of the test program the system operated without incident and without significant operator interaction. The predominant operator activity consisted of regular recording of process conditions. However, the crucible binding incident highlights the need for design review of rotating crucible plasma systems.

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- Improvements in the gas scrubber system are required. Test data indicate that the RIF-2 scrubbers are ineffective in removing acid gases, particulates and other compounds. Improved scrubbers will reduce the load on the HEPA filters, helping the system to meet regulatory emission limits.
- The data available to date indicate that the effluent streams from a PLASMOX<sup>®</sup> unit treating NSCMP neutralent will likely be disposed as non-hazardous wastes. Confirmation of these results are in progress. A report addendum will be issued when this data is available.
- Insufficient analytical data is currently available to complete a phosphorus balance. Additional analyses have been initiated in order to estimate the disposition of phosphorus throughout the RIF-2 system. A report addendum will be issued when this data is available. Preliminary analysis of the solid material collected from the reactor exit piping indicate that the phosphorus may be plating out inside the system. A preliminary analysis of stack gas sampling condensate from Test Run GB-1 also indicate that phosphorus may be leaving the system via the stack gas. Chemical analysis of material deposited in the reactor exit piping after Test Run HD-1 indicated the presence of silicon, phosphorus and chlorine. The mechanism for the formation of this deposited material is unclear. The presence of silicon and phosphorus in the deposited solid material demonstrates that silicon is depleted from the slag and deposits in the system and that phosphorus also deposits in the system. The high concentration of particulate material in the stack gas is likely a result of slag volatilization and condensation – no data has been supplied that would support the assumption that particulate loading results from refractory conditioning. It is possible that entrainment and deposition of the slag material is attributable to specific system design features, such as insufficient disengagement space in the reactor. Testing of other plasma systems should investigate the design issues that may contribute to this effect.
- Test data have shown that NO<sub>x</sub> emissions, carbon monoxide, hydrocarbons and organic Hazardous Air Pollutants are all below regulatory limits. Testing of other plasma systems should focus on the ability of properly designed off-gas treatment systems to reduce particulate and metal concentrations to below MACT limits. Similarly, future tests of plasma systems that utilize a ROC or secondary combustor should focus on combustor process control to reduce potential formation of dioxins/furans. The PLASMOX<sup>®</sup> system tests could not confirm whether detectable levels of dioxins/furans were a result of equipment contamination.
- The MGC electrode and torch designs provide significant improvements over competing water-cooled electrode torch designs in terms of reliability and safety. Although the PLASMOX® electrode and torch reduce the risk of releasing torch cooling water that could cause pressure excursions, it would be prudent to include expansion tanks for any NSCMP applications. In addition, significant improvements to the efficacy of the unit can be made by optimizing the feed locations, designing the ROC for the specific feeds

being processed and by sizing the Off-Gas Treatment System for processing of liquid feeds.

Based on these conclusions, the following are recommended:

- It is recommended that a plasma system be tested at the pilot scale on NSCMP liquid and solid waste streams. These tests should be prototypical of full-scale operation.
- Pilot scale testing of a plasma system should investigate materials of construction impacts
  of the various NSCMP waste streams and the long-term effect of, and mechanism of
  deposition of material in the system.
- Additional data to support permitting of a plasma system should be collected during pilot scale testing.
- A survey of plasma system technology providers indicates that there are more than a
  dozen potential competitive sources of plasma systems. Additional testing of plasma
  technology should capitalize on the availability of competing designs to address the
  technical issues identified in these tests.

### EVALUATION OF THE LIMITED ENGINEERING SCALE TESTING OF THE PLASMOX® TECHNOLOGY TO TREAT CHEMICAL WARFARE MATERIEL

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### Evaluation of the Limited Engineering Scale Testing of the PLASMOX® Technology to Treat Chemical Warfare Materiel

### 1.0 INTRODUCTION

This report evaluates the results of the Limited Engineering Scale Tests of the Burns and Roe/MGC PLASMOX<sup>®</sup> Plasma (PLASMOX<sup>®</sup>) process. Stone & Webster conducted these tests to assess the operability of the PLASMOX<sup>®</sup> process when treating NSCMP feeds. This included an assessment of the stability and throughput capabilities of the system when treating NSCMP liquid waste streams.

MGC conducted the tests under a subcontract to Burns & Roe Enterprises, Inc. (Burns & Roe) at the MGC PLASMOX® facility in Switzerland. The tests were conducted as part of the systemization of the PLASMOX® Research and Integration Facility 2 (RIF-2) unit during the weeks of January 8 through January 19, 2001. Only existing equipment and instrumentation was used to collect test data so as not to interfere with the MGC-planned shipment of the unit to Albania in February 2001.

This section discusses the objectives of the Limited Scale Engineering Tests and the Evaluation Criteria that Stone & Webster developed to evaluate the test's performance. Section 2 of this report provides a summary of Stone & Webster's technology evaluation efforts and the rationale for selecting PLASMOX® for testing. Section 3 describes the PLASMOX® RIF-2 unit that was tested. Section 4 describes all of the test runs completed as part of the Limited Engineering Scale Testing. The results of the tests are presented in Sections 5 and 6. Section 5 includes a discussion of the operability of the PLASMOX® unit. Section 6 presents the analytical results of the samples collected during testing. Sections 7 and 8 include the test conclusions and recommendations for further action.

### 1.1 Test Objectives

The principal objective of the Limited Engineering Scale Tests was to assess the operability of the PLASMOX<sup>®</sup> plasma system to treat NSCMP neutralents. In developing the test plan for the tests, Stone & Webster specified five test objectives:

- 1) Demonstrate maximum throughput for each feed.
- 2) Demonstrate continuous, stable operability of the PLASMOX® System for each feed type.
- 3) Demonstrate ability to process feeds such that process effluents can be disposed in a RCRA facility without the need for additional treatment.
- 4) Determine the fate of phosphorus.
- 5) Obtain engineering data to support preliminary design.

Burns & Roe/MGC developed the Limited Engineering Test Plan and Sampling and Analysis Plan based on the above objectives. Each of the objectives is discussed below.

### 1.1.1 Maximum Throughput

The ability of the PLASMOX<sup>®</sup> plasma system to process liquid wastes containing a high percentage of water was a concern of Stone & Webster's. According to initial discussions with Burns & Roe and MGC<sup>1</sup> high concentrations of water in the feed stream "slows down the through put of the system." According to Burns and Roe and MGC, the bottleneck in the PLASMOX<sup>®</sup> process is the Rapid Oxidation Chamber (ROC) where synthesis gases generated in the plasma reactor are oxidized.

The size of a unit to treat the expected quantities of NSCMP wastes generated at a facility, such as Pine Bluff Arsenal, will depend largely on the liquid throughput capacity of the unit. Therefore, demonstration of the maximum liquid throughput capacity of the PLASMOX® unit was a goal of the tests, especially given the concerns about water-containing feeds previously raised by MGC and Burns and Roe.

### 1.1.2 Continuous Stable Operation

The second objective of the Limited Engineering Scale Tests of the PLASMOX<sup>®</sup> system was to demonstrate continuous stable operation. Demonstration of a maximum feed rate provides a basis for scale-up of the system. However, if the feed material is not sufficiently processed at this rate, or the system operates erratically at these conditions, then it can not be considered a viable scaling point. Stable operation also provides a subjective measure of the operability of the process and demonstrates how the control system responds to upset conditions.

### 1.1.3 Disposable Effluents

Data from the Limited Engineering Scale Tests may be used to develop designs to support permit applications. Therefore, a third objective of the tests was to characterize the process effluent streams and compare the composition of these streams against applicable disposal criteria.

### **1.1.4** Fate of Phosphorus

Theoretical calculations and some experimental data have shown that high temperature processes that operate under reducing conditions have a higher probability of producing phosphine when treating phosphorus-containing compounds. This possibility is reduced in the case of the PLASMOX® system because of the oxidation that occurs in the Rapid Oxidation Chamber, downstream of the reducing plasma reactor. However, because of the toxicity of phosphine, a fourth objective of the tests was to document the fate of phosphorus.

### 1.1.5 Provide Engineering Data

The primary reason for conducting Limited Engineering Scale Tests was to identify and quantify key engineering parameters needed to support preliminary engineering and environmental permitting. In addition to performance data, Stone & Webster also evaluated the operation of the PLASMOX® system during the tests in terms of worker safety, training of operators, maintainability and operability.

### 1.2 Evaluation Criteria

Associated with each of the five test objectives are evaluation criteria that were used to assess the effectiveness of the tests. The evaluation criteria and associated objectives are discussed below.

### **1.2.1** Evaluation of Maximum Throughput

Maximum throughput will be evaluated based on a simulant feed rate, selected by Burns and Roe/MGC, at which the RIF-2 unit can operate safely and effectively. Maximum throughput will be used to evaluate the scale of a PLASMOX<sup>®</sup> plasma unit that would be appropriate for a particular NSCMP application. The following conditions must be met for a feed rate to be considered maximum throughput:

- Stable operation, as defined below.
- Effluent streams meet disposal criteria, as defined below.

### **1.2.2** Evaluation of Continuous Stable Operation

To evaluate the continuous stable operation of the RIF-2 unit during processing of the simulant feeds, the following criteria were applied:

- Operation of the unit with all systems controlled and no system functions overridden for the duration of the test period.
- No emergency shut-down of the system.

### 1.2.3 Evaluation of Disposability of Effluents

To evaluate the disposability of effluents from the PLASMOX® system, the following criteria were applied:

- Is the TOC content of all liquid waste streams less than 25 ppm?
- Do the liquid waste streams meet disposal requirements from a Federal Wastewater Treatment Facility?
- Can the solid waste generated be disposed of at a RCRA facility?

### **1.2.4** Evaluation of Fate of Phosphorus

This criterion applies only to the GB simulant feed stream. To evaluate this objective analytical data was used to develop a rough material balance for phosphorus.

### 1.2.5 Evaluation of Engineering Data

To evaluate the availability of engineering data, the following questions were posed:

- What are the system's operating characteristics?
- What are the unit's system safety engineering safeguards?
- What is the reliability, availability and maintainability of the unit?
- How easy will it be to permit a unit?

### 2.0 BACKGROUND

The U.S. Army Program Manager for Chemical Demilitarization (PMCD) established the NSCMP with the mission to provide centralized management and direction to the Department of Defense for the disposal of non-stockpile chemical materiel in a safe, environmentally sound and cost effective manner. The NSCMP includes five categories of chemical warfare materiel (CWM): binary chemical weapons; former production facilities; miscellaneous CWM; recovered chemical weapons; and buried CWM. Substantial differences exist between CWM in the Stockpile and Non-Stockpile programs. Whereas the stockpiled CWM is present in larger quantities, non-stockpile CWM encompasses a greater variety of materiel with far more physical configurations and agent-fill types. The variety, locations and deteriorated physical condition of non-stockpile CWM pose unique requirements for treatment systems.

To support accomplishment of its mission, the NSCMP developed an Overarching Research Plan<sup>2</sup> (ORP) which establishes the goals, requirements, and approaches for evaluating and developing technologies for the safe and efficient disposal of non-stockpile CWM. The ORP identifies systems that NSCMP has and is continuing to develop to meet its mission goals. The ORP also identifies additional needs and associated schedule to support accomplishment of these goals.

To meet these needs, NSCMP has identified several additional systems for application to non-stockpile CWM based on the results of technology evaluations and demonstration testing performed as part of the PMCD Alternative Technologies and Approaches Program (ATAP) and the Assembled Chemical Weapons Assessment Program (ACWAP).

In 1999 NSCMP tasked Stone & Webster with identifying and evaluating technologies that could be used to destroy NSCMP neutralent waste streams. Stone & Webster prepared a request for technology information packages that was published by the government in a Commerce Business Daily (CBD) Announcement on May 13, 1999, soliciting interest from technology providers. Additionally, in an effort to broaden the search beyond the normal CBD audience, Stone & Webster sent 150 letters to universities and technology companies, advertising the solicitation. The process resulted in thirteen technology information packages submitted by technology providers.

The NSCMP also requested that the technology information packages be evaluated by an independent Technology Evaluation Panel (TEP). Stone & Webster organized a panel of seven (7) members selected from academia and industry with expertise in biology, chemistry, chemical and mechanical engineering, technology evaluation and implementation and permitting. Stone & Webster also invited five (5) Citizen Members to participate in the evaluation process along with the Technology Evaluation Panel.

In its report to NSCMP, the independent TEP recommended that the PLASMOX<sup>®</sup> system be tested for its ability to treat NSCMP neutralent streams.<sup>3</sup>

The results of the Assembled Chemical Weapons Assessment Program (ACWAP) test of the Startech Plasma Waste Converter (PWC)<sup>4</sup> had previously confirmed that a plasma-based process could destroy agent hydrolysate. However, ACWAP did not recommend the

technology for destruction of agent because of concerns about potential steam excursions. The NSCMP evaluation of numerous plasma technologies<sup>5</sup>, however, provided sufficient confidence in the technology's ability to treat NSCMP neutralent wastes.

Based on the TEP recommendation, NSCMP (Edward F. Doyle, Alternative Systems Demonstration and Evaluation Group Leader) directed Stone & Webster to evaluate the potential of using the Startech Plasma Waste Converter (PWC) unit for demonstration testing on NSCMP feeds before pursuing testing of the PLASMOX® system. NSCMP had obtained the PWC from ACWAP after completion of the ACWAP Phase I testing. Stone & Webster put together an independent panel of experts from industry and government to evaluate the PWC unit. After a review of the PWC testing history, site visits and discussions with Startech personnel, the panel recommended that the PWC unit not be used for demonstration testing.

The evaluation of the PWC identified the use of water cooled metal electrode torches in plasma reactors as a critical safety concern for application of plasma to NSCMP wastes. The performance of metal electrode torches in the PLASMOX® system was therefore of particular concern as Stone & Webster developed the evaluation criteria for the PLASMOX® tests.

### 3.0 SYSTEM DESCRIPTION

The Limited Engineering Scale Testing of PLASMOX® to treat chemical warfare material was performed at MGC Plasma, AG facilities in Müttenz, Switzerland using a PLASMOX® RIF 2 transportable unit. This unit, which has been utilized for similar tests as well as actual destruction of chemical agents in Europe, had just undergone significant modifications in preparation for being shipped to Albania to support the destruction of chemical warfare material for the Albanian Ministry of Defense.

The RIF 2 unit consists of three transportable "modules" as shown in Figure 3-1<sup>7</sup>. These are:

- **Plasma Reactor Skid** which includes the plasma reactor vessel, a Rapid Oxidation Chamber (ROC), hydraulic power system and associated electrical power and distribution panels.
- Power Supply and Control System Skid which includes the main control console, power supply and transformer, electrical distribution panel and process gas interface manifolds.
- Off-Gas Treatment System Skid which includes a quench subsystem, 2-stage scrubber system, gas heater, HEPA filter and a fan for maintaining system negative pressure.

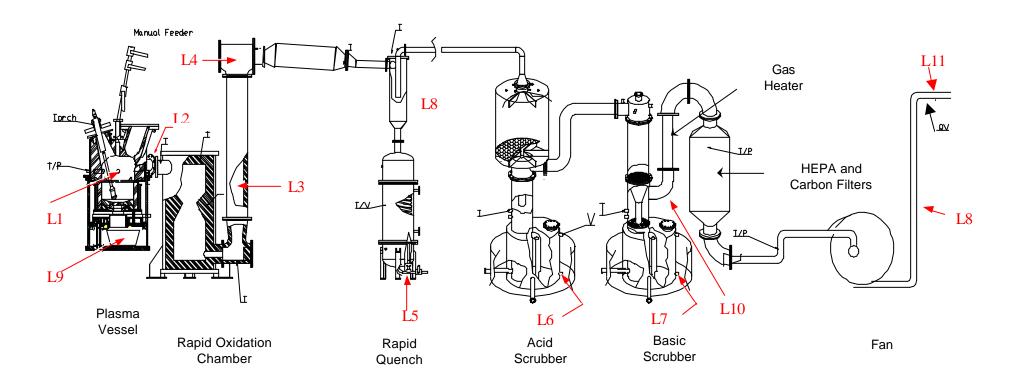
The system was installed within an existing MGC building that provided all necessary support functions.

According to MGC, the RIF 2 System has a maximum throughput capacity of 50 kg/hr (25 kg/hr organic-equivalent), depending on the feed material composition. The entire system is designed as a closed system operating at sub atmospheric pressure (approximately 950 mbar). Each of the major system components is discussed below.

### 3.1 Plasma Reactor Vessel and Plasma Torch

The PLASMOX plasma reactor vessel is a closed hearth which consists of two separable sections. The top reactor section, "lid," contains the plasma torch port, feed/exhaust ports and ports for monitoring equipment such as video cameras and temperature measurements. The feed ports consist of one 5 X 5 cm port for solids/bulk material and one liquid lance for liquids. Figure 3-2 shows the inside section of the reactor top. This section slides horizontally to expose the lower reactor section. The inside diameter of the lid is 23.6 inches (60 cm); the inside depth is 17.1 inches (43.5 cm).

Figure 3-1 - PLASMOX System Layout



The lower reactor section contains the rotating crucible hearth. Figure 3-3 is a photograph of the lower reactor section. The refractory-lined hearth is not cooled. The refractory lining is a brick material composed of an aluminum oxide and chromium oxide composite. Before each campaign, the crucible is loaded with a glass/limestone slag. The slag is arranged so that a portion of the carbon bottom plate is exposed. The exposed portion of the crucible bottom acts as an electrode to strike the torch. Once the slag is melted, it becomes conductive.

The crucible rotates at variable speeds in order for the centrifugal force to prevent the molten slag material from being discharged through a center tap hole. When tapping of the molten slag material is desired, the rotational speed is lowered to permit the melt to flow to the center of the crucible and to be discharged through the tap hole orifice. A slag collection mold is located beneath the reactor to catch the slag and residue being discharged from the crucible (see Figure 5-1). The center tap hole orifice can be closed if the plasma reactor will be operated with a large molten bath in a batch mode. The inside diameter of the rotating crucible is 23.6 inches (60 cm); the inside depth is 11 inches (28 cm).

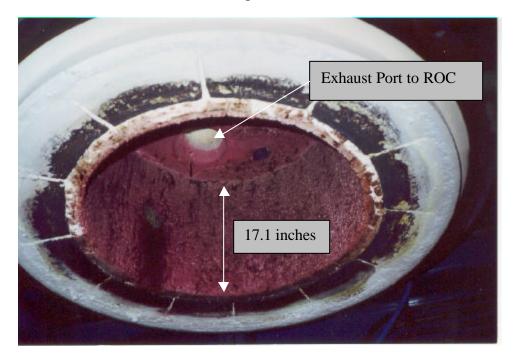


Figure 3-2 - Top Reactor Section of Reactor Vessel

The plasma reactor operates under a slight negative pressure to contain the gaseous emissions and direct them into the rapid oxidation chamber and the gas treatment system. The plasma reactor is water-cooled by a system separate from the plasma torch cooling system.

The transferred arc plasma torch is a hollow electrode plasma torch with a copper electrode that can be operated at up to 200 kW. Both the torch and electrode are MGC proprietary designs. Typical voltages are 250 - 450 V at currents from 250 - 450 A. The torch is equipped with a separate water cooling system. The location of the torch within the chamber

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is controlled hydraulically from the control panel. The torch is easily removed to allow for the periodic change of the electrode.

"Cold Start" of the system is accomplished using helium and a high voltage torch starter. After startup the system is run using nitrogen  $(N_2)$ . At times, oxygen is also fed into the Plasma Reactor Vessel to control particulate "fog" common during operations under reducing conditions.

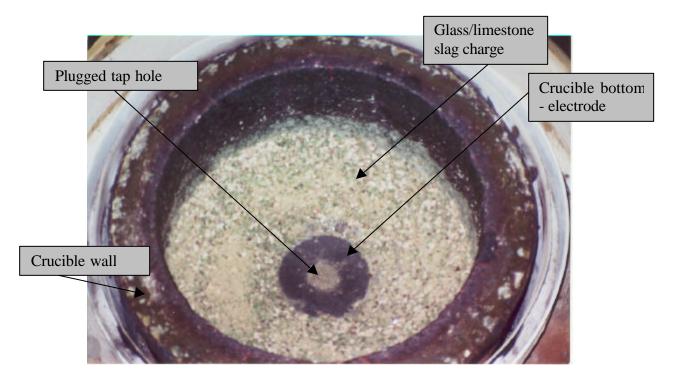


Figure 3-3 - Lower Reactor Section and Rotating Crucible of Reactor Vessel

### 3.2 Rapid Oxidation Chamber

Exhaust gases from the Plasma Reactor Vessel flow into the Rapid Oxidation Chamber (ROC) which serves to completely destroy remaining volatile organic compounds (VOCs). It is equipped with nozzles near the top, middle and bottom of the chamber to allow oxygen or air to be blown into the chamber, a propane fired burner at the top of the chamber and temperature controllers to maintain a chamber temperature above 1,000°C. The reactor exhaust gases enter the top of the ROC near the propane burner and flow downward. System throughput is controlled to maintain the residence time of the gases generated for more than 2 seconds to ensure complete oxidation. The ROC is a 2m³ (70.6 ft³) carbon steel vessel with a ceramic refractory. Throughput capacity is rated at 300Nm³/hr (10,593 SCF/hr).

MGC designed and fabricated a new ROC currently installed on the RIF-2 for the destruction of picric acid. The picric acid comprises a large percentage of the chemical warfare materiel stockpile in Albania. The Swiss Army has contracted MGC to use the RIF-2 unit to destroy

this chemical stockpile. Because MGC will pump the picric acid directly into the ROC for destruction, the chamber incorporates a combustion control system that is independent of the Plasma Reactor Vessel and Off-Gas Treatment System controls.

### 3.3 Off-Gas Treatment System

Major components of the Off-Gas Treatment System include a rapid quench subsystem, an acid scrubber, a basic scrubber, heater, HEPA filter and a fan which maintains the entire system upstream of the fan at a sub atmospheric pressure.

Rapid Quench Subsystem – The rapid quench subsystem is a closed direct spray quench system consisting of a horizontal stainless steel quench chamber, cyclone moisture separator and quench water tank (320L). During normal operation, high temperature off-gases from the ROC enter the quench chamber and are cooled by co-current direct water injection (spray). The cooled off-gas then enters the cyclone where excess water is removed for collection in the quench water tank. Water in the quench water tank is cooled by a closed cooling water system (cooling coils) and is reused as the source of water for the quench subsystem.

**Acid/Basic Scrubbers** – As off-gases from the quench subsystem enter the acid scrubber, water from a re-circulation pump is injected into the scrubber throat as a spray into the gas stream. After the air/water mixture leaves the throat, the larger drops and particulates fall into the sump where the water and solids are collected. The off-gas stream then flows through the basic scrubber to the HEPA and carbon filters.

**Heater** – A blower draws ambient air across an electric coil heater. The heated air mixes with the scrubber exhaust to keep the stack gas at a temperature above its condensation temperature. Before the start of the first test run, MGC installed a carbon filter over the air suction to prevent volatile compounds from being drawn from the test facility building into the stack gas and adversely impact the test results.

**HEPA Filter** – The HEPA filter was not installed in the filter housing for the demonstration testing.

**Fan** – The entire system is maintained at a negative pressure by a standard axial fan rated at 900Nm<sup>3</sup> (output) at a total static head of 10Kpa. Motor is rated at 11KW, 400V, 3 hp.

### 3.4 Miscellaneous Systems

Power to the RIF 2 torch is supplied at 200kW by an air cooled, 12 pulse thyristor rectifier operating at 380V, 50Hz. The following ancillary systems are also provided at MGC facilities in Müttenz to support the RIF 2 operation:

- Electrical Power 450kVA (3 phase, 380V, 50 Hz)
- Emergency Power 100kVA Generator
- Gas Supply Sufficient bottled gas supply to provide the following:
  - Nitrogen 20 Nm<sup>3</sup>/hr

- Oxygen  $-20 \text{ Nm}^3/\text{hr}$
- Propane 10 Nm<sup>3</sup>/hr
- Compressed Air 10 Nm<sup>3</sup>/hr
- Helium for torch start only
- Cooling Water 4.7 L/sec (20m³/hr at 6 bar)

### 3.5 Process Instrumentation

The RIF 2 System is provided with instrumentation and an on-line gas analyzer for continuous on-line monitoring and recording of O<sub>2</sub>, NO/NO<sub>2</sub>, CO/CO<sub>2</sub>, SO<sub>2</sub>, hydrocarbons, temperature, pressure and air flows (volume) at the monitoring points indicated on the P&ID, included in Appendix B.

Samples of the process stream for analysis in accordance with the Sampling and Analysis Plan were collected at the following sampling points as indicated in Figure 3-1. These locations are:

•	L4 Rapid Oxidation Chamber Exhaust	gas sample
•	L5 Quench Water Tank	water sample
•	L6, L7 Acid and Basic Scrubbers	waste water sample
•	Basins Acid and Basic Scrubbers	sludge sample (no solids from tests)
•	L8 System Exhaust (Stack)	gas sample
•	L9 Slag Extraction Chamber	melt sample

### 4.0 TEST DESCRIPTIONS

This section presents descriptions of the PLASMOX RIF-2 plasma unit tests conducted during January 2001. The test descriptions include a discussion of the simulants used in the tests, descriptions of each test run and the sampling analyses conducted..

### 4.1 Simulants

Two NSCMP neutralent simulants were tested during the six test runs. Both feed streams simulated Monoethanolamine (MEA)-based Munitions Management Device (MMD) neutralents. The composition of the two simulant streams are shown in Table 4-1.

	Component in Simulant	Chemical Formula	Simulant MW	Wt% in Simulant
H in MEA Neutralent	MEA	C <sub>2</sub> H <sub>7</sub> NO	61	83
	Water	$H_2O$	18	10
	HC1	HC1	36.5	7
GB in MEA Neutralent	MEA	C <sub>2</sub> H <sub>7</sub> NO	61	40
	Water	$H_2O$	18	52
	DMMP	C <sub>3</sub> H <sub>9</sub> O <sub>3</sub> P	124.07	8

**Table 4-1 Simulant Compositions** 

Both the H in MEA Neutralent and GB in MEA Neutralent simulants are based on a 10:1 volume ratio of reagent to chemical agent. The reagent used for mustard detoxification is 90% MEA and 10% water; the GB reagent is 45% MEA and 55% water. 9

According to NSCMP estimates, the majority of CWM that will be treated in the Non-Stockpile program contain mustard. Any technology selected for the treatment of NSCMP neutralents must be capable of safely and successfully processing mustard neutralents. A smaller percentage of NSCMP CWM contains GB. The MGC PLASMOX process has been demonstrated in the treatment of metal parts and therefore metal parts were not included in the test program. Demilitarization Protective Ensemble (DPE) suits will be a significant feed material for an NSCMP treatment system. The ACWAP Startech Plasma tests demonstrated the ability of plasma systems to treat mixed solid wastes included DPE. DPE were not included as part of the PLASMOX Limited Engineering tests because the RIF-2 unit was not equipped to feed full DPE suits.

A primary objective of the Limited Engineering Scale tests was to evaluate the ability of the PLASMOX system to process organic feed streams containing high concentrations of water. The Startech ACWAP test results had previously demonstrated that plasma technologies can destroy caustic hydrolysate streams from the treatment of mustard and nerve agent. The

simulant recipes for the PLASMOX tests represent typical MEA and water concentrations in NSCMP neutralent wastes.

Note that the mustard in MEA simulant does not contain sulfur which, according to NSCMP, is estimated to make up less than 2 molar percent of the H in MEA neutralent. Note also that the GB in MEA simulant does not contain fluorine, which is estimated to contain less than 1.5 molar percent fluorine. The simulants represent the feed constituents of primary concern, namely MEA and water in both neutralent feeds and the C-P bond in the GB neutralent stream. Fluorine was also left out of the GB neutralent simulant because of concerns for potential corrosion – the RIF-2 unit was scheduled to be transported to Albania at the end of the Limited Engineering Scale tests and corrosion damage from fluorine, if it occurred, could jeopardize the Albanian campaign.

### 4.2 Test Run Descriptions

The Limited Engineering Scale Testing of the PLASMOX process consisted of six discreet test runs at the MGC facilities in Muttenz, Switzerland between January 12 and January 19, 2001. In addition to the six test runs, MGC conducted a Work-Up run to validate the operation of the liquid feed lance. No analytical data were collected from the Work-Up run.

The Burns and Roe/MGC test plan proposed eight 6-hour test runs – four tests with GB in MEA Neutralent simulant and four with H in MEA Neutralent simulant. The length and number of test runs were based on a nominal 8-hour workday, a 40-hour work-week and the availability of the unit for NSCMP testing for two weeks. In addition to the eight test runs, Burns and Roe/MGC also proposed a work-up run to prove-out the liquid lance operation.

The test plan specified sampling and analysis of liquid and solid streams from all eight test runs and gas stream sampling during one GB Neutralent simulant run and one HD Neutralent simulant run. Burns and Roe subcontracted the sampling to Dr. Graf, AG of Bern, Switzerland and analytical work to Dr. Meyer AG, Bern, Switzerland, an accredited EN45'001 laboratory (Swiss Accreditation).

Table 4-2 summarises the actual test runs completed using simulant feeds. A work-up run to fine-tune the operation of the liquid feed lance was completed on January 11, 2001 using a water/MEA mixture. As shown in Table 4-2, three out of four planned 6-hour runs using GB Neutralent simulant(GB-1, GB-2, GB-3) were completed. In addition, the final two mustard neutralent simulant test runs (HD-3/4) were combined into a single 12-hour test run. The reasons for these deviations from the test plan are described in the individual test descriptions below.

TEST	GB-1	GB-2	GB-3	HD-1	HD-2	HD-3/4
Date	12 Jan 01	13 Jan 01	15 Jan 01	16 Jan 01	17 Jan 01	18 Jan 01
Start Feed	1834 Hrs.	1500 Hrs.	1430 Hrs.	1636 Hrs.	1358 Hrs.	1400 Hrs.
Stop Feed	0119 Hrs. 13 Jan 01	2100 Hrs.	2030 Hrs.	2236 Hrs.	2036 Hrs.	0200 Hrs. 19 Jan 01

**Table 4-2 - Test Summary** 

Each test run began with an initial charge of slag of approximately 10-15 kg of a glass and limestone mixture consisting of 10 parts crushed bottle glass (cullet) and 1 part limestone. Once the initial charge of slag is placed in the crucible reactor, the reactor is sealed and ROC burner is started. The ROC requires approximately two hours to heat up before the plasma torch is ignited and the initial charge of slag is melted. Approximately three hours are required to melt the initial charge and to complete heating of the ROC to an operating temperature of 1,050°C in the mid-section of the ROC. Once the initial slag charge is melted and the ROC is at temperature, the liquid feed is begun. During liquid feeding, additional slag is added to the reactor chamber.

It is the MGC PLASMOX normal operating procedure to feed oxygen to the plasma reactor vessel during operation of the RIF-2 unit. The reason for the oxygen, according to Burns and Roe/MGC, is to reduce soot formation and allow the operator to view the reaction chamber at all times. The use of oxygen in the reactor vessel is not documented in the Test Plan supplied to Stone & Webster prior to beginning the tests and is contrary to the intent of operating in an inert or reducing atmosphere. Burns and Roe/MGC informed Stone & Webster about the use of oxygen after all but the last test run had been completed. For this test run, Test Run HD-3/4, the system was operated with no oxygen to the plasma reactor vessel. The data from the final H simulant run will be compared against the other runs in order to assess the effect of oxygen.

### 4.2.1 Work-up Test Run

The Limited Engineering Scale Tests of the PLASMOX process for NSCMP neutralents was the first time that liquid feeds had been treated in the RIF-2 unit. MGC fabricated a liquid feed lance for this series of tests. The lance was placed into one of the open access ports in the lid of the plasma reactor, approximately one foot from the reactor outlet port in the side wall of the reactor. MGC fitted the lance with a metering feed pump that withdrew simulant feed from a nearby tank and fed the liquid into the plasma reactor.

MGC intended to spray the liquid feed into the reactor by mixing air with the neutralent and aspirating the material into the reactor chamber. On January 10, 2001, MGC began testing the liquid feed lance with a 50/50 water/MEA mixture. The plasma reactor and ROC were both at temperature (1350°C and 1050°C) when the feed rate was increased from 2 L/hr to 4

L/hr. At this point the torch failed and water began flowing from the torch onto the molten slag. The system reacted to the steam build up in the plasma reactor, but was limited by pipe diameter restriction after the quench. The RIF-2 blower control system automatically responded to the increase in pressure caused by the steam by pulling more gas through the Off-Gas System and ROC. The diameter of the pipe after the Rapid Quench restricted the flow of gas such that the system blower pulled a vacuum on the first scrubber vessel. The heat and vacuum caused the vessel to warp. The torch was removed within 5 minutes of the event and the vessel re-sealed.

The plasma reactor did not overpressure and would likely have been quickly evacuated of excess steam if not for the pipe diameter restriction. Upon inspection of the failed electrode, MGC concluded that the pulsing air used to aspirate the liquid in the feed lance had either forced solid material into the electrode, forming a high resistance area, or the pulsing pressure disrupted the spiraling movement of the plasma arc within the electrode. The net effect was excessive arcing in a focused area of the electrode. MGC modified the liquid feed system by eliminating the use of air to aspirate the feed. There were no further electrode failures during the remainder of test runs; all five electrodes used in the remaining tests showed even wear.

The following day, January 11, Burns and Roe/MGC completed two Work-Up Test run campaigns to prove-out the liquid feed lance. After the experience with aspirating air, MGC modified the feed system to feed liquid only. Campaign 1 used a 50/50 water/MEA mixture. Campaign 2 used the GB neutralent simulant of MEA/DMMP/water. During each campaign, MGC started the feed rate at 2 L/hr and increased the feed rate 2 L/hr every 20 minutes to a maximum of 12 L/hr. The RIF-2 unit operated smoothly and without interruption for two full hours during both campaigns.

### 4.2.2 GB Neutralent Simulant Test Runs

The GB Test Runs were originally scheduled to begin January 11. According to the original schedule, the gas sampling subcontractor, Dr. Graf AG made arrangements to be present at the site on January 12 to collect gas samples during the second GB Test Run, GB-2. Because of the torch failure during the first Work-Up run, the GB runs did not begin until January 12. Consequently, gas samples were collected during test run GB-1. Liquid and solid samples were collected from all three GB Test Runs and analyzed. Continuous monitoring of stack effluents was also conducted by Dr. Graf AG during all three GB Test Runs.

Table 4-3 summarizes all three GB Test Runs. The table provides all of the significant operating data, including slag charges, slag recovery, start and stop times for each of the major operating systems, the flow of nitrogen plasma gas and oxygen to the plasma reactor and ROC and liquid feed rate. The table also summarizes the total test time during which simulant was fed to the reactor, total liquid fed over the course of the test, the number of hours of torch operation, average torch operating parameters, ROC setpoint and the operating range of reactor vessel pressure.

### 4.2.2.1 Test Run GB-1

Test Run GB-1 was run on January 12, 2001. Dr. Graf AG collected gas samples during this test run. Dr. Graf AG believe that a leak in one of the gas sampling ports caused an anomaly in some of the on-line test data collected during this run. Figure 4-1 is a graph of the on-line test data collected during GB-1. The data shown on Figure 4-1 represent conditions in the exhaust gas downstream of the blower. The reported oxygen and carbon dioxide content of this stream includes the effects of heated dilution air bled into the stack gas upstream of the HEPA filters. The organic carbon, carbon monoxide and nitrogen oxide values have been adjusted for the dilution air effect and are therefore representative of conditions at the ROC. These data are designated as "undiluted" in Figure 4-1.

The wide swings in exhaust gas concentrations between the start of the ROC at 13:00 hrs. and the start of liquid feed at 18:30 hrs. are believe to be the result of numerous adjustments to the system including propane feed and oxygen to the ROC and the opening and closing of the port used for the liquid feed lance. The exact cause of system perturbations are assumed to be documented in the operator logs. Stone & Webster has requested the logs from Burns and Roe in order to confirm the cause of the variations.

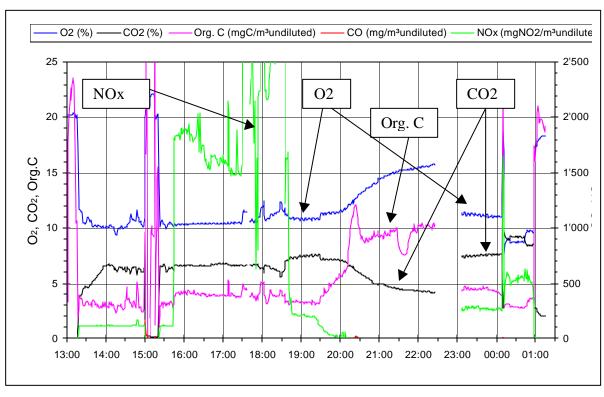


Figure 4-1 - On-Line Stack Data from GB-1<sup>10</sup>

The GB Neutralent Simulant was fed to the plasma reactor at an initial rate of 8 liters per hour (L/hr) beginning at exactly 18:34 hrs. (Table 4-3) and then increased to 12.3 L/hr. This

higher feed rate, demonstrated during the Work-Up Run, was maintained for the remainder of the GB-1 Test Run. Figure 4-1 shows that once the feed was introduced to the plasma reactor (18:34 hrs.) the on-line analyzers showed a sharp decrease in  $NO_X$  concentration in the exhaust and a sharp increase in organic carbon. At the same time there was a gradual increase in oxygen concentration and decrease in carbon dioxide. It was determined that the cause of this anomaly was an in-leakage of air into the heated filter on the sampling line. At approximately 22:30 hrs. the filter was removed, inspected and replaced. When on-line sampling of the exhaust resumed 30 minutes later, the oxygen,  $CO_2$ , organic carbon and  $NO_X$  returned to levels consistent with levels immediately after the introduction of feed. (Compare values at 19:00 hrs. with values at 23:00 hrs.)

The disturbance at 0:10 hrs. on January 13 was caused when the ROC burner was turned off for several minutes.

Overall, Test Run GB-1 was completed without major interruptions. The plasma torch logged more than 9.5 hours of operation at an average power loading of 149 kW. A total of 82.8 liters of liquid simulant was processed during the test run at an average feed rate of 12.3 L/hr over 6 hours and 45 minutes. Reactor vessel pressure was maintained between 11.2 and 24.0 mbar. All systems operated as designed.

### 4.2.2.2 Test Run GB-2

Test Run GB-2 was conducted on Saturday January 13, 2001. Because of the delay in starting the test runs, Stone & Webster requested, and Burns and Roe/MGC agreed to combine Test Runs GB-2 and GB-3 into a continuous 12 hour run. Test Run GB-4 would then be scheduled for Monday January 15, followed by HD-1 on January 16, HD-2 – the gas sampling run – on January 17 and a combined HD-3/4 12-hour run on January 18-19.

As shown on Table 4-3, Test Run GB-2 began at 07:30 hrs. with the start of the ROC. The plasma torch was struck at 11:30 hrs. and the GB Neutralent Simulant feed was introduced at 12.3 L/hr at 15:00 hrs. Nitrogen plasma gas feed to the torch was 6.7 Nm³/hr., the same rate as used for Test Run GB-1. The oxygen flow to the reactor for this run was decreased from the previous test run.

Figure 4-2 shows the on-line stack data collected during Test Run GB-2. The collection of data was begun when the feed was introduced. For this run Dr. Graf AG replaced the filter on the stack gas sample port. As can be seen in Figure 4-2, there was no air in-leakage. All parameters were stable throughout the entire run.

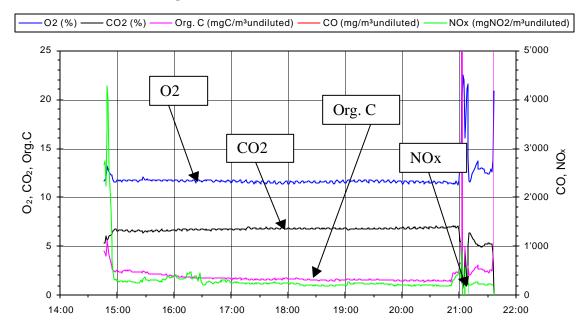


Figure 4-2 On-Line Stack Data from GB-2<sup>11</sup>

At approximately 11:00 hrs. the operators heard a grinding noise coming from the plasma reactor centrifuge. Burns and Roe/MGC decided at this point to halt the GB-2 Test Run in order to minimize any potential damage to the reactor and centrifuge. It was not apparent what the cause of the problem was. Burns and Roe/MGC could not pour the molten slag because of the possibility of damaging the centrifuge or crucible; something appeared to be lodged between the rim of the crucible and the reactor lid or wall. The RIF-2 was shut down and allowed to cool over the weekend.

On Monday morning, January 15 the reactor was opened and inspected. Molten slag had deposited on the rim of the crucible and built up so as to fill the space between the crucible and the lid of the reactor. Because no slag was poured, the cooled slag had to be chiseled out of the crucible after the system had cooled.

Although Test Run GB-2 ran for 6 hours without major interruptions, the deposition of slag disrupted the continuous operation of the unit. The plasma torch logged more than 9.5 hours of operation at an average power loading of 142 kW. A total of 73.8 liters of liquid simulant was processed during the test run at an average feed rate of 12.3 L/hr over 6 hours. Reactor vessel pressure was maintained between 15.2 and 23.9 mbar. With the exception of the slag feed and crucible, all systems operated as designed.

Table 4-3 - GB Test Runs Summary 12

	GB-1	GB-2	GB-3
Date of Test –2001	January 12	January 13	January 15
Initial Slag Charge (kg) (1)	11.0	11.0	16.5
Additional Slag Feed (kg) (1)	22.0	33.0	22.0
Start ROC	13:16 h	07:30 h	10:30 h
Start Plasma Torch	15:44 h	11:31 h	11:43 h
Start Liquid Feed	18:34 h	15:00 h	14:30 h
Nitrogen Plasma Gas Flow (Nm <sup>3</sup> /h)	6.7	6.7	6.7
Oxygen Supply to Reactor (Nm <sup>3</sup> /h)	6.4	4.8	4.8/2.4
Oxygen Supply to ROC (Nm <sup>3</sup> /h)	4.8	4.8	4.8/2.4
Liquid Feed Rate (L/h)	Ramp to 12.3	12.3	12.3
Stop Liquid Feed	01:19	21:00 h	20:30 h
Total Liquid Processed (L)	82.8	73.8	73.8
Slag Pour Time	01:24 h	No pour (2)	20:45 h
Stop Plasma Torch	01:27 h	21:04 h	20:50 h
Stop ROC	01:30 h	21:36 h	20:54 h
Slag Poured (kg)	32.5	0	35.0
Slag removed from Reactor vessel (kg)	0	41.5	1
Slag remaining in Reactor Vessel (kg) (for	1	0	0
Slag Recovered	98%	94%	94%
Total Feeding Time (h:mm)	6:45	6:00	6:00
Total Liquid Feed (l)	82.8	73.8	73.8
Total Plasma Torch Operating Time (h:mm)	9:47	9:33(3)	9:07(3)
Average Torch Power (kW)	149	142	145
Average Torch Voltage (V)	424	419	413
Average Torch Current (A)	351	338	351
Setpoint ROC (°C)	1053	1053	1053
Negative pressure Reactor Vessel (mbar)	11.2 - 24.0	15.2 - 23.9	9.3 – 19.0

**Notes:** 1. Blend of 10 parts bottle glass and 1 part lime stone. During heat treatment glass looses weight (mainly moisture) and limestone decomposes to CaO and CO<sub>2</sub> (50 % weight loss).

- 2. Centrifuge stalling due to slag deposition in the gap (lid to centrifuge upper rim) → new: screw feeder displaced to lid center
- 3. Same torch used for both Test Run GB-2 and GB-3. Total Time on Electrode was 18hrs 40min.

### 4.2.2.3 Test Run GB-3

Test Run GB-3, a 6-hour test, was completed without incident on January 15, 2001. Because of the delay in starting the test runs and the inability to complete a 12-hr test run on January 13, Stone & Webster decided to eliminate Test Run GB-4 in order to provide time to complete a full test run with the high-chloride H in MEA Neutralent simulant feed stream prior to full gas sampling, which was scheduled for January 17.

As shown on Table 4-3, Test Run GB-3 began at 10:30 hrs. with the start of the ROC. The plasma torch was struck at 11:40 hrs. and the GB Neutralent Simulant feed was introduced at 12.3 L/hr at 14:30 hrs. Over the 6-hour test, 73.8 liters of simulant feed were processed. The same torch and electrode used for Test Run GB-2 was used for Test Run GB-3. The torch operated successfully for a total of 18 hours and 40 minutes. Burns and Roe/MGC recommend that the torch electrode be replaced after 20 hours.

Figure 4-3 shows the on-line stack data collected during Test Run GB-3. The collection of data was begun when the feed was introduced. As can be seen in Figure 4-3, all parameters were stable throughout the entire run. Test Run GB-3 was completed without major interruptions. The plasma torch logged more than 9 hours of operation at an average power loading of 145 kW. Reactor vessel pressure was maintained between 9.3 and 19.0 mbar. All systems operated as designed.

CO2 (%) Org. C (mgC/m³undiluted) CO (mg/m³undiluted) NOx (mgNO2/m³undiluted) O2 (%) 25 5'000 Org. C 4'000 20 O2D<sub>2</sub>, CO<sub>2</sub>, Org.C CO<sub>2</sub> 3'000 15 **NO**x 2'000 10 5 1'000 0 14:00 15:00 16:00 17:00 18:00 19:00 20:00 21:00

Figure 4-3 - On-Line Stack Data from GB-3<sup>13</sup>

### 4.2.3 H Neutralent Simulant Test Runs

The first H Test Run, HD-1, was scheduled to begin January 16 so that a full run with the H in MEA Neutralent simulant could be run before the sampling of gas streams was conducted on January 17. Stone & Webster decided that it was important that a full test run be completed before the gas sampling run based on the experience of Test Run GB-1. According to this schedule, the gas sampling subcontractor, Dr. Graf AG would be present at the site on January 17 to collect gas samples during the second H Test Run, HD-2. Liquid and solid samples were collected from all three H Test Runs and analyzed. Continuous monitoring of stack effluents was also conducted by Dr. Graf AG during all three H Test Runs.

Table 4-4 summarizes all three H Test Runs. The table provides all of the significant operating data, including slag charges, slag recovery, start and stop times for each of the major operating systems, the flow of nitrogen plasma gas and oxygen to the plasma reactor and ROC and liquid feed rate. The table also summarizes the total test time during which simulant was fed to the reactor, total liquid processed for each test, the number of hours of torch operation, average torch operating parameters, ROC setpoint and the operating range of reactor vessel pressure.

### 4.2.3.1 Test Run HD-1

Test Run HD-1 was run on January 16, 2001. Figure 4-4 is a graph of the on-line test data collected during Test Run HD-1. The data shown on Figure 4-4 represent conditions in the exhaust gas downstream of the blower. The reported oxygen and carbon dioxide content of this stream includes the effects of heated dilution air bled into the stack gas upstream of the HEPA filters. The organic carbon, carbon monoxide and nitrogen oxide values have been adjusted for the dilution air effect and are therefore representative of conditions at the ROC. These data are designated as "undiluted" in Figure 4-4.

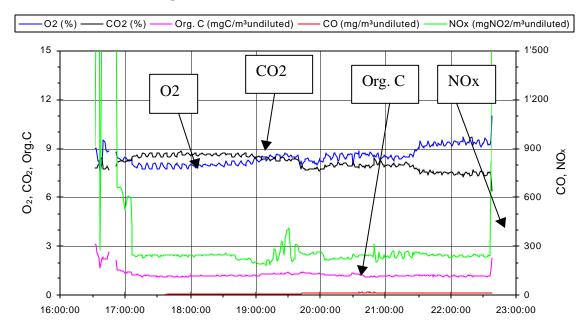


Figure 4-4 - On-Line Stack Data from HD-1<sup>14</sup>

Because no Work-Up Run had been conducted with the H Neutralent Simulant, the liquid simulant was fed to the plasma reactor beginning at 16:30 hrs. at an initial rate of 2 L/hr. Every 20 minutes the feed rate was increased by 2 liters per hour to a test feed rate of 6 L/hr. This feed rate was maintained for the remainder of the HD-1 Test Run.

Because of the higher organic content of the H in MEA Neutralent simulant, Burns and Roe/MGC had to operate the ROC differently than in the GB test runs. As shown in Table 4-4, oxidant was supplied to the ROC via both oxygen and air. During the GB test runs, no air was supplied to the ROC. Air was used to dilute and attenuate the heat-up of the refractory in the ROC. The use of air and oxygen may have impacted the disturbance at approximately 19:30 hrs. when the relative concentrations of oxygen and carbon dioxide in the stack gas reversed. Stone & Webster has requested that Burns and Roe provide a copy of the operators log book so that the cause of this change can be established.

Over the 6-hour test, 34.5 liters of simulant feed were processed. The torch operated successfully for 8 hours and 36 minutes. The collection of continuous stack data was begun when the feed was introduced at approximately 16:30 hrs. As can be seen in Figure 4-4, all parameters were relatively stable throughout the entire run. Test Run HD-1 was completed without major interruptions. The plasma torch operated at an average power loading of 148 kW. Reactor vessel pressure was maintained between 10.1 and 24.1 mbar. All systems operated as designed.

When the reactor had cooled and opened to reset the limestone and glass slag charge for Test Run HD-2, the crucible walls appeared blacker and more stained than after the GB runs.

#### 4.2.3.2 Test Run HD-2

Test Run HD-2 was run on January 17, 2001. Dr. Graf AG collected gas samples during this test run. Figure 4-5 is a graph of the on-line stack test data collected during the course of run HD-2. The data shown on Figure 4-5 represent conditions in the exhaust gas downstream of the blower. The reported oxygen and carbon dioxide content of this stream includes the effects of heated dilution air bled into the stack gas upstream of the HEPA filters. The organic carbon, carbon monoxide and nitrogen oxide values have been adjusted for the dilution air effect and are therefore representative of conditions at the ROC. These data are designated as "undiluted" in Figure 4-5.

Simulant feed to the unit was begun at approximately 14:00 hrs. and was stopped at approximately 20:30 hrs. The causes of the disturbances at 14:30 hrs. and 17:30 hrs. are not clear. Stone and Webster assumes that the system perturbations were caused by changes in system parameters, such as the switch from air and oxygen in the ROC to oxygen only. Stone & Webster has requested that Burns and Roe provide a copy of the operators log book so that the origins of these variations can be established.

Over the 6.5-hour test, 39.3 liters of simulant feed were processed. The torch operated successfully for 10 hours. As can be seen in Figure 4-5, all parameters were relatively stable throughout most of the run, with the exception of the periods already noted. Test Run HD-2 was completed without major interruptions. The plasma torch operated at an average power loading of 134 kW. Reactor vessel pressure was maintained between 13.0 and 23.0 mbar. All systems operated as designed.

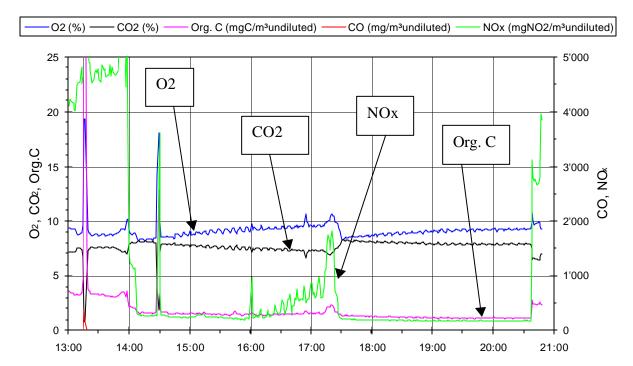


Figure 4-5 - On-Line Stack Data from HD-2<sup>15</sup>

### 4.2.3.3 Test Run HD-3/4

Test Run HD-3/4 was begun at approximately 14:00 hrs. on January 18, 2001 and completed 12 hours later at approximately 02:15 hrs. on January 19. Test Run HD-3/4 was a combined 12 hour test run.

During the first six hours of the test, simulant was fed to the plasma reactor at a rate of 6 L/hr, consistent with the two previous HD Test Runs. Stone & Webster had requested that Burns and Roe/MGC demonstrate the unit at what they believed was the maximum throughput. Burns and Roe/MGC had opted initially to operate the test unit at what they considered conservative throughputs in order to protect the RIF-2 equipment, and specifically the ROC, for the Albanian Campaign. They did agree to gradually increase the simulant feed rate for the last half of the final HD run to the maximum throughput.

Table 4-4 - H Test Runs Summary 16

	HD-1	HD-2	HD-3/4
Date of Test –2001	January 16	January 17	January 18
Initial Slag Charge (kg) (1)	16.5	11.0	11.0
Additional Slag Feed (kg) (1)	11.0	11.0	11.0
Start ROC	13:40 h	09:41 h	10:00 h
Start Plasma Torch	14:20 h	10:53 h	12:00 h
Start Liquid Feed	16:36 h	13:58 h	14:00 h
Nitrogen Plasma Gas Flow (Nm <sup>3</sup> /h)	6.7	6.7	6.7
Oxygen Supply to Reactor (Nm <sup>3</sup> /h)	4.8	5.6	0
Oxygen Supply to ROC (Nm³/h)	3.2 (+ 18.0 air)	18.0 air (later in run 4 O <sub>2</sub> , air off)	
Liquid Feed Rate (L/h)	6.0 (ramp 2, 4, 6)	6.0	10.8 / 13.0 (ramp 6. 8. 10)
Stop Liquid Feed	22:36 h	20:36 h	02:00 h
Total Liquid Processed (L)	34.5	39.3	122.8
Slag Pour Time	22:52 h	20:50 h	02:10 h
Stop Plasma Torch	22:56 h	20:54 h	02:13 h
Stop ROC	23:00 h	20:58 h	02:16 h
Slag Poured (kg)	19.0	20.5	16.0
Slag removed from Reactor vessel (kg) (after test)	0	0	2
Slag remaining in Reactor Vessel (kg) (for next run, approx)	6	0	0
Slag Recovered	91%	93%	81%
Total Feeding Time (h:mm)	6:00	6:38	12:00
Total Liquid Feed (l)	34.5	39.3	122.8
Total Plasma Torch Operating Time (h:mm)	8:36	10:01	14:13
Average Torch Power (kW)	148	134	142
Average Torch Voltage (V)	416	416	423
Average Torch Current (A)	356	321	335
Setpoint ROC (°C)	1053	1044	1053
Negative pressure Reactor Vesel (mbar)	10.1 - 24.1	13.0 – 23 .0	14.6 – 23.9

**Notes:** 1. Blend of 10 parts bottle glass and 1 part lime stone. During heat treatment glass looses weight (mainly moisture) and limestone decomposes to CaO and  $CO_2$  (50 % weight loss).

After the first six hours of Test Run HD-3/4 the feed rate was increased to 8 L/hr, 10 L/hr and eventually 13 L/hr. Figure 4-6 is a graph of the on-line stack test data collected during the course of run HD-3/4. The data shown on Figure 4-6 represent conditions in the exhaust gas downstream of the blower. The reported oxygen and carbon dioxide content of this stream includes the effects of heated dilution air bled into the stack gas upstream of the HEPA filters. The organic carbon, carbon monoxide and nitrogen oxide values have been adjusted for the dilution air effect and are therefore representative of conditions at the ROC. These data are designated as "undiluted" in Figure 4-6.

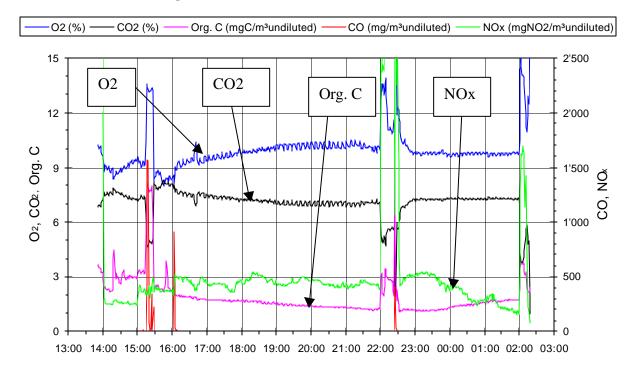


Figure 4-6 - On-Line Stack Data from HD-3/4

Simulant feed to the unit was begun at 14:00 hrs. and was stopped at approximately 02:00 hrs the following day. The causes of the disturbances between 15:00 and 16:00 hrs. are not clear. Stone and Webster assumes that the system perturbations were caused by changes in system parameters, such as the adjustment of air and oxygen in the ROC. The disturbance at approximately 22:00 hrs. was caused by the adjustment of the simulant feed rate. Stone & Webster has requested that Burns and Roe provide a copy of the operators log book so that the origins of these variations can be established.

Over the 12-hour test, 122.8 liters of simulant feed were processed. The torch operated successfully for 14.25 hours. As can be seen in Figure 4-6, all parameters were relatively stable throughout most of the run, with the exception of the periods already noted. Test Run HD-3/4 was completed without interruption. The plasma torch operated at an average power loading of 142 kW. Reactor vessel pressure was maintained between 14.6 and 23.9 mbar. All systems operated as designed.

### 4.3 Sampling and Analysis

The Burns and Roe Test Plan<sup>17</sup> specified collection of samples from the effluent streams from the RIF-2 unit for analysis. Three categories of streams were sampled:

- 1) Solid and liquid grab samples from the completion of all test runs;
- 2) Continuous on-line analysis of gas samples during all test runs; and
- 3) Grab samples from specific gas streams during one GB and one H test run respectively.

The Sampling and Analysis Plan was finalized with the Sampling Subcontractor, Dr. Graf AG during a meeting on January 9, 2001. Tables 4-5 and 4-6 summarize the grab samples finalized for the GB and H Test Runs, respectively, during the January 9 meeting. 18 Characterization of the solid and liquid streams was conducted in order to asses whether these streams could be disposed of without further treatment and to assist in the development of material balance data. The continuous on-line measurements provided data on the stability of the process and whether gaseous effluents from the PLASMOX system would require treatment prior to be discharged to the atmosphere. Analysis of the gas grab samples provided information on the ability of the PLASMOX system to destroy the materials of concern and provided additional data concerning the need for post treatment of gaseous effluents.

### 4.3.1 Solid and Liquid Grab Samples

Grab samples were collected from the vessel melt and scrubber water. The Test Plan specified collection and analysis of scrubber sludge, however, no sludge was generated during any of the test runs.

#### **4.3.1.1** Vessel Melt

The Burns and Roe Test Plan specified the collection of two melt grab samples for each simulant demonstration test. The grab samples are collected from the slag extraction chamber (L9 in Figure 3-1) and analyzed in accordance with Methods for Analysis shown in Tables 4-5 and 4-6. The first melt sample was to be taken of the "clean" initial melt after system start up and prior to initiating simulant feed. The second melt sample was to be taken after completion of the four test runs of each simulant.

Instead of collecting one sample before initiating feed and a second sample after completion of all simulant runs, samples were collected after each of the individual test runs. A total of three melt samples were collected and analyzed for the GB Test Runs and four samples were collected from the H Neutralent simulant Test Runs.

These grab samples consisted of a glass-like silica and were in the form of solid chunks. In order to perform the required analysis these solid chunks are crushed by a standard sample preparation method into a relatively fine powder. This powder was further treated (digested, extracted, etc.) in accordance with appropriate techniques listed for the analyte of interest and the type of solid powdered matrix provided. Complete analyses of the melt samples are included in Appendix A.

Table 4-5 – Proposed Sampling and Analysis Matrix for GB Test Runs 19

MATRIX	ANALYTE	# OF SAMPLES	SAMPLE METHOD(S)	PRESERVATIVE	SAMPLE SIZE	TEST METHOD(S)
Stack Gas	Aldehydes	1	Mod SW-846:0011	4°C	NA	HPLC, VDI 3862. Part 2
(L8)	Hydrocyanic acid	1	Impinger Train	4°C	NA	SW-846: 9213
	Metals	1	Filter	pH<2	NA	EMPA SOP 1884. VDI 3868, Part 1
	Phosphorus, Aluminum	1	Filters	PH<2	NA	VDI 3868; ICP-AES, VS-132, VS-157
	Particulates	1	Filter	NA	NA	EMPA SOP 1533, VDI 2066, Part 1, 2, 7
	MEA(C <sub>2</sub> H <sub>7</sub> NO)	1	Sorbent Tube	NA	NA	GC-FID, GC-MS
	DMMP(C <sub>3</sub> H <sub>9</sub> O <sub>3</sub> P)	1	Mod SW-846:0031	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	NA	GC-FID, GC-MS
	SVOCs	1	Mod SW-846:0031	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	NA	GC-FID, GC-MS
	VOCs	1	Mod SW-846:0030	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	NA	GC-FID, GC-MS
Scrubber Liquid	Aldehydes	4	Grab	$Na_2S_2O_3$ , $4^{\circ}C$	250 ml	HPLC, VDI 3862. Part 2
& Quench Water	TOC	4	Grab	4°C	40 ml	EMPA SOP 1883, VDI 3481 Part 1,3 6
(Note: Separate	Cyanide total	4	Grab	4°C	500 ml	DIN EN 38405, Sect 13
each scrubber and	Phosphate	4	Grab	4°C	50 ml	DIN EN ISO 10304-1
quench tank)	Nitrate	4	Grab	4°C	100 ml	DIN EN ISO 10304-1
(L5 & L6, L7)	MEA(C <sub>2</sub> H <sub>7</sub> NO)	4	Grab	4°C	2ml	GC-FID, GC-MS
	DMMP(C <sub>3</sub> H <sub>9</sub> O <sub>3</sub> P)	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	1000ml	GC-FID, GC-MS
quench tank) N (L5 & L6, L7) N  E S	SVOCs	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	1000 1	GC-FID, GC-MS
	VOCs	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	5 ml	GC-FID, GC-MS
Scrubber Sludge	Total Weight	4	Grab			Density/Volume
Samples, one, from	TOC	4	Grab	4°C	40 ml	EMPA SOP 1883, VDI 3481 Part 1,3 6
each scrubber basin)	Metals	4	Grab	pH<2	200 ml	EMPA SOP 1884, VDI 3868, Part 1
	Phosphorous total	4	Grab	4°C	50ml	DIN EN ISO 10304-1
	MEA(C <sub>2</sub> H <sub>7</sub> NO)	4	Grab	4°C	2ml	GC-FID, GC-MS
	DMMP(C <sub>3</sub> H <sub>9</sub> O <sub>3</sub> P)	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	1000ml	GC-FID, GC-MS
	TCLP	4	Grab	4°C	2000 ml	Swiss TVA 814.600
Vessel Melt	Total Weight	2	Grab	NA		Density/Volume
Vessel Melt	TOC	2	Grab	NA	10g	EMPA SOP 1883, VDI 3481 Part 1,3 6
	Metals	2	Grab	NA	200g	EMPA SOP 1884, VDI 3868, Part 1
	Phosphorous total	2	Grab	NA	50g	DIN EN ISO 10304-1
	TCLP	2	Grab	NA	2000g	Swiss TVA 814.600

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Table 4-6 – Proposed Sampling and Analysis Matrix for H Test Runs <sup>20</sup>

MATRIX	ANALYTE	# OF SAMPLES	SAMPLE METHOD(S)	PRESERVATIVE	SAMPLE SIZE	TEST METHOD(S)
Stack Gas	Aldehydes	1	Mod SW-846:0011	4°C	NA	HPLC, VDI 3862. Part 2
	Dioxins-Furans	1	Sorbent Tube	NA	NA	EMPA SOP 1891, SN EN 1948 Part 1-3, GC-MS
	Hydrochloric acid	1	Impinger Train	4°C	NA	EMPA SOP 1887, VDI 3480 Part 1
	Hydrocyanic acid	1	Impinger Train	4°C	NA	SW-846: 9213
	Metals	1	Filter	pH<2	NA	EMPA SOP 1884. VDI 3868, Part 1
	Particulates	1	Filter	NA	NA	EMPA SOP 1533, VDI 2066, Part 1, 2, 7
	MEA(C <sub>2</sub> H <sub>7</sub> NO)	1	Sorbent Tube	NA	NA	GC-FID, GC-MS
	SVOCs	1	Mod SW-846:0031	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	NA	GC-FID, GC-MS
	VOCs	1	Mod SW-846:0030	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	NA	GC-FID, GC-MS
Scrubber Liquid	Aldehydes	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	250 ml	HPLC, VDI 3862. Pg 2
& Quench Water	TOC	4	Grab	4°C	40 ml	EMPA SOP 1883, VDI 3481 Part 1,3 6
(Note: Separate	Chloride	4	Grab	NA	50 ml	EMPA SOP 1887, VDI 3480 Part 1
Samples, one, from	Cyanide total	4	Grab	4°C	500 ml	DIN EN 38405, Sect 13
each scrubber and	Dioxins-Furans	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	1000 ml	GC-MS
quench tank)	Nitrate	4	Grab	4°C	100 ml	DIN EN ISO 10304-1
(L5 & L6, L7)	MEA(C <sub>2</sub> H <sub>7</sub> NO)	4	Grab	4°C	2ml	GC-FID, GC-MS
	SVOCs	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	1000 1	GC-FID, GC-MS
	VOCs	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	5 ml	GC-FID, GC-MS
Scrubber Sludge	Total Weight	4	Grab			Density/Volume
(Note: Separate	Dioxins-Furans	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	1000 ml	GC-MS
Samples, one, from	TOC	4	Grab	4°C	40 ml	EMPA SOP 1883, VDI 3481 Part 1,3 6
each scrubber basin)	Metals	4	Grab	pH<2	200 ml	EMPA SOP 1884, VDI 3868, Part 1
	PCBs	4	Grab	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , 4°C	1000 ml	GC-MS
	MEA(C <sub>2</sub> H <sub>7</sub> NO)	4	Grab	4°C	2ml	GC-FID, GC-MS
	TCLP	4	Grab	4°C	2000 ml	Swiss TVA 814.600
Rapid Oxidation Chamber Outlet (L4)	Dioxins-Furans	1	Sorbent Tube	NA	NA	EMPA SOP 1891, SN EN 1948 Part 1-3, GC-MS
Vessel Melt	Total Weight	2	Grab	NA		Density/Volume
(L9)	TOC	2	Grab	NA	10g	EMPA SOP 1883, VDI 3481 Part 1,3 6
	Metals	2	Grab	NA	200g	EMPA SOP 1884, VDI 3868, Part 1
	TCLP	2	Grab	NA	2000g	Swiss TVA 814.600

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### 4.3.1.2 Scrubber Liquid and Quench Water

Grab samples of the quench water and scrubber liquids were planned to be collected at Locations L5, L6 and L7 (in Figure 3-1) for analysis in accordance with the Methods in Tables 4-5 and 4-6. The Test Plan proposed collection of liquid from each of the three sample locations at the conclusion of each of the four test runs for each simulant. During testing, an additional sample was collected from the fresh water in the scrubbers before beginning the first test run of each simulant. At the end of Test Run GB-3 and collection of the liquid samples, the scrubbers were drained and refilled with fresh water for the H Simulant test runs.

### 4.3.1.3 Scrubber Sludge

Grab samples of the scrubber sludge were proposed for analysis following the methods in Tables 4-5 and 4-6. However, no precipitates or sludge were generated and, therefore, no sludge samples were collected.

### 4.3.2 Continuous On-Line Stack Analysis

The following five parameters were measured continuously in the stack, downstream of the system fan, during all tests:

- Oxygen by paramagnetic detector
- Carbon dioxide by infrared detector
- Carbon monoxide by infrared detector
- Nitrogen oxides by chemical illuminescence detector
- Hydrocarbons by flame ionization detector

Analyses of the system showed that the stack gas was diluted with heated ambient air to prevent condensation in the gas duct. Therefore heated, cleaned air was added before the guard filter box (HEPA filter). To be able to correct for this dilution air, an additional oxygen measurement was installed behind the scrubber and before the dilution air inlet. The oxygen content upstream of the dilution air was used to adjust the concentrations of CO, NO<sub>x</sub> and hydrocarbons to non-dilution conditions.

### 4.3.3 Stack Grab Samples

A grab sample from the system stack, downstream of the fan (Location L8 in Figure 3-1) was planned to be collected for analysis during one of the GB Neutralent simulant test runs. The Test Plan proposed analysis of the sample in accordance with the Methods in Tables 4-5.

## Grab sampling during test GB-1 from stack gas

- Aldehydes (one sample, 1 h sampling time)
- Hydrocyanic acid (one sample, 3 h sampling time)
- Metals: copper, chromium, iron and zinc; particle bound and volatile parts, (one sample, 3 h sampling time)

- Particulates (one sample, 3 h sampling time)
- Monoethanolamine (one sample, 2 h sampling time)
- Dimethyl-methylphosphonate (one sample, 3 h sampling time)
- Identification of SVOC's and VOC's (by GC-MS-Fingerprint)

Although specified in the final Sampling and Analysis Plan on January 9, the stack gas was not analyzed for phosphorus. This discrepancy was not made known to Stone & Webster until after the data analysis report was delivered in March 2001. At that time Burns and Roe indicated that the phosphorus analysis would be provided. Burns and Roe/MGC subsequently directed Dr. Graf AG to re-analyze the sample for phosphorus and aluminum. However, the original sample from Test Run GB-1 had been consumed as part of the original analysis. Instead, Dr. Graf AG analyzed a stack gas sample from Test Run HD-2. The results of this analysis were delivered April 27, 2001 in an Addendum report.

### Grab sampling during test GB-2 between reactor and ROC

During the operation of Test Run GB-2 Stone & Webster noted that the location of the liquid feed nozzle in the lid of the plasma reactor, relative to the reactor exhaust, increase the possibility of feed material bypassing the reaction zone. Therefore, Stone & Webster requested that a gas sample be collected between the plasma reactor and the ROC and that the sample be analyzed for MEA. Dr. Graf AG was able to complete this collection during Test Run GB-2.

- Monoethanolamine (one sample, 1 h sampling time)

#### Grab sampling during test HD-2 from stack gas

- Particulates (one sample, 3 h sampling time)
- Metals: copper, chromium, iron and zinc; particle bound and volatile parts (one sample, 3 h sampling time)
- Phosphorus (particle bound and volatile parts, one sample, 3 h sampling time)
- Aluminum (particle bound part only, one sample, 3 h sampling time)
- Hydrocyanic acid (one sample, 3 h sampling time)
- Hydrochloric acid (one sample, 3 h sampling time)
- Aldehydes (one sample, 1 h sampling time)
- Monoethanolamine (one sample, 2 h sampling time)
- Dioxins and furans (one sample, 4 6 h sampling time)
- Identification of SVOC's and VOC's (by GC-MS-Fingerprint)

# Grab sampling during test HD-2 between reactor and ROC

- Monoethanolamine (one sample, 1 h sampling time)

# Grab sampling during test HD-2 between ROC and quench

- Dioxins and furans (one sample, 4 - 6 h sampling time)

#### 5.0 OPERABILITY

This section presents Stone & Webster's comments on the design and operation of the RIF-2 unit as observed during the PLASMOX<sup>®</sup> Limited Engineering Scale tests. Comments on the operation of the major equipment items, standard operating procedures, process safety and worker safety are provided.

The operation of the following systems were observed and are commented on below:

- Plasma Reactor
- Plasma Arc Electrode and Torch
- Rapid Oxidation Chamber
- Off-Gas Treatment System

In addition to these systems, Stone & Webster also observed the Burns and Roe/MGC use of Standard Operating Procedures (SOP), and their approach to training and safety.

#### 5.1 Plasma Reactor

The rotating crucible has an outside diameter of 30.5 inches (77.5 cm) and a height of 18.5 inches (47 cm). The inside dimensions are 23.6 inches (60 cm) diameter and 11 inches (28 cm) deep. The crucible weighs approximately 1,500 pounds (680 kg). According to Burns and Roe/MGC, the changeover time for a crucible is approximately two hours. Stone & Webster did not observe a changeover during the test period. The crucible is designed to rotate up to 50 revolutions per minute (rpm). Typically, in this test run series, the crucible rotated in a counter-clockwise direction at a rate of one rotation every 2 seconds (30 revolutions per minute).

The internal volume of the crucible is 2.75 cubic feet  $(0.08 \text{ m}^3)$ . For each of the test runs, up to 36 pounds (16.5 kg) of crushed glass cullet and limestone (10 parts glass to 1 part limestone) were evenly distributed in the bottom of the crucible. The distributed material prior to the start of a test run is shown in the bottom of the crucible in Figure 3-2. Figure 5-1 is a photograph of the cooled slag material poured from the crucible at the end of a typical test run. During each test run additional slag material (up to 73 pounds [33 kg]) was added to the molten bath. When melted, the molten slag acts as a heat sink for the thermal energy provided by the torch. The thermal energy supplied by the torch and stored in the molten slag breaks the chemical bonds of the liquid neutralent simulant. The plasma torch is typically located 2 to 3 inches (5-7.5 cm) from the rotating melt bed. Therefore, as shown in Figure 5-2, the plasma arc impinges on the surface of the crucible and the melt.

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Figure 5-1 - Collected Slag in Mold Below Reactor

### **5.1.1** Deposition of Material

Deposition of material was observed at two times during the test. The first occurred at the end of the GB-2 Test Run, when material collected on the rim of the crucible, causing the crucible to bind up. As a result, the test run was stopped at this point, without pouring the molten slag.

The second observation of deposition was in the outlet of the Plasma Reactor. This occurred at least twice during the test runs: after the GB-2 Test Run; and after the HD-1 Test Run. Material from the throat of the outlet was collected for analysis after the HD-1 Test Run.

#### 5.1.1.1 Crucible Edge

After approximately six hours into the GB-2 test on 15 Jan 01 the rotating crucible began to emit a noticeable grinding sound. Instead of risking damage to the equipment Burns and Roe/MGC decided to stop the test run after six hours instead of continuing the planned 12-hr. run. Inspection of the reactor, after the system had cooled, revealed that the top edge of the crucible was covered with a hardened layer of glassy slag material. This slag had filled up the small clearance space between the rotating crucible and the bottom of the upper reactor. As the crucible rotated, the glassy layer was grinding against the stationary top reactor section (see Figure 5-3).

The glassy slag buildup on the top edge of the crucible could have resulted from one or more of the following conditions:

• Some glass cullet added to the crucible during the test runs could have fallen on the top edge of the crucible.

- The plasma torch could have vaporized some glass cullet which deposited on the cooler surfaces within the clearance space.
- Molten slag could have splashed onto the top edge of the crucible when additional slag material was fed into the crucible.



Figure 5-2 - Typical Plasma Torch in Operation

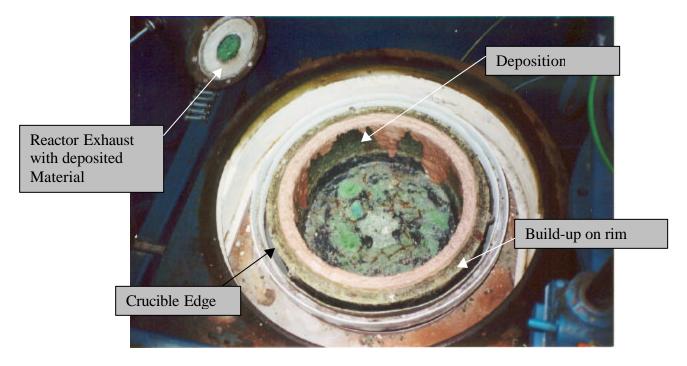


Figure 5-3 - Glass Slag Build-up on Crucible Rim

Figure 5-3 is a photograph showing the deposited molten slag on the rim of the crucible and inside walls. Burns and Roe/MGC believed that the cause of the build-up was a result of glass and limestone feed port being located directly above the rim of the crucible. According to Burns and Roe/MGC, this would not normally have been problematic and in fact, it was not a problem during GB-1. Two factors are believed to have contributed to the build-up of slag on the rim of the crucible during GB-2:

- 1) The crucible used for the NSCMP tests was designed for the Albanian campaign and had a thicker wall at the rim than the typical MGC crucible; and
- 2) The operators inadvertently added too much slag to the reactor after the initial charge had been melted.

The typical MGC crucible has a tapered wall at the rim. The wall thickness is approximately 3.5 inches and tapers back to approximately 2 inches over the top 2 inches (approximately) of the crucible wall. Based on the available information, MGC decided to forego the tapering of the crucible rim for the crucibles fabricated for the Albania Campaign. Normally, the slag feed port on the reactor lid is positioned over the tapered (inside) edge of the crucible rim. Any slag material that might impact the crucible rim likely flows off the edge and into the reactor. A level rim limits the ability of molten slag to move off the rim. During GB-2, MGC believes that slag material fell onto the hot rim, melted and remained, eventually building up and binding on the lid of the reactor.

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The likely reason why build-up of slag on the rim did not bind the crucible during Test Run GB-1 is because the amount of slag added to GB-2 was 50% greater than what was added during GB-1. As shown in Table 4-3, 33 kg of slag was added during GB-2 after the initial charge of 11 kg had melted. Only 22 kg of additional slag was added during GB-1. According to Burns and Roe/MGC, the operator misread the procedure. Instead of adding slag for a total charge of 33 kg (11 initial plus 22 additional), an additional 33 kg of slag was added. The additional slag combined with the location of the feed is believed to have caused build-up and eventual binding.

The hardened layer of slag on the rim of the crucible was removed with a grinding machine and the hardened slag in the crucible was chiseled out. Prior to beginning Test Run GB-3, The RIF-2 operators moved the location of the slag feed port to a location on the lid closer to the center of the crucible, by switching it with the video camera. No further build-up of slag on the rim of the crucible was experienced during the remainder of the tests.

This problem is indicative of the need to pre-test and evaluate the reactor design and procedures to preclude such problems during live neutralent destruction campaigns. The incident also exposed a potential design problem with rotating crucible plasma reactors. Because the reactor melt can not be poured in the current RIF-2 design if the crucible is not spinning at high revolutions, any loss of power or gearing to the crucible turntable would create a situation similar to what occurred at the end of Test Run GB-2. There is a potential for only partially reacted material to be entrained in the melt and for the melt to be chiseled out of the crucible. Further design considerations are required to address the potential of worker exposure through this route.

#### **5.1.1.2** Reactor Outlet

During the removal of the slag buildup on the top edge of the crucible after Test Run GB-2, it was noted that the gas exhaust pipe was clogged with a green residue (see Figure 5-3). A similar situation occurred after Test Run HD-1. The cause or impact of the deposition of material at this location is uncertain. This deposition could have been caused by one or more of the following conditions:

- Vaporized glass or molten slag could have deposited in the cooler gas outlet pipe. This assumption is supported by the evidence of deposition on the "cooler" walls of the crucible.
- Because the liquid feed port on the RIF-2 unit is located near the reactor outlet, some of the liquid neutralent could have bypassed the reaction chamber, and the "relatively" cooler liquid could have caused preferential cooling at the exhaust. This would have had the effect of causing increased deposition at the throat of the reactor as observed.

Stone & Webster requested that a sample of this material be submitted to the laboratory and analyzed. The results of this analysis indicate that silicon and phosphorus are deposited in the system piping. (See Section 6.1.4.)

### **5.1.2** Vessel Geometry Issues

The combined top reactor section and rotating crucible have inside dimensions of 23.6 inches (60 cm) in diameter and 28.1 inches (71 cm) in height. The interior reactor volume of the crucible is 7.11 ft3 (0.2 m3). The general experience in the plasma industry has shown that for plasma torch power levels of approximately 150 kW, the inside diameters of plasma reactors generally do not exceed 24 inches, with reactor volumes less than 13 cubic feet. Using these criteria the PLASMOX® plasma reactor vessel conforms with standard industry practice.

As previously mentioned, the location of the liquid feed port on the RIF-2 on the top of the plasma reactor is adjacent to the exhaust port. This proximity increases the risk of material bypassing the hottest portions of the plasma reactor. Bypassing material may also have contributed to residue buildup in the gas outlet pipe as discussed previously. Samples were collected from the reactor outlet, between the reactor and the ROC and analyzed for MEA during a GB simulant and an HD simulant test run. Analysis of the samples detected no MEA. These results seem to indicate that organic material in the simulants was processed in the plasma reactor. Regardless of these results, the liquid feed port on a commercial plasma unit to treat NSCMP wastes should be located so as to minimize the risk of material bypassing the reaction zone.

#### 5.2 Plasma Arc Torch and Electrode

The water-cooled copper plasma torch electrode used in the PLASMOX® system is operated in the transferred arc mode up to a power level of 200 kW. The electrode is the only consumable in the PLASMOX® torch. Metal, water-cooled plasma torches are subject to erratic failure, which could allow large amounts of water to be discharged into the hot processing reactor in a very short period of time. This could result in a violent steam excursion within the processing vessel. This possibility of discharging water into the reactor presents a potential reliability and safety hazard. If the feedstock materials are chemical agent neutralents, the resulting pressure spike could result in a release of hazardous gases and liquids out of the processing reactor, into the surrounding work areas, and potentially into the atmosphere. The following paragraphs discuss PLASMOX® torch electrode life, reliability and safety with regard to these issues.

# 5.2.1 PLASMOX® Plasma Torch Design

Figure 5-4 is a photograph of the 200 kW plasma torch. The torch body has approximate dimensions of 3.5 inches (8.9 cm) diameter and 9.2 inches (23.4 cm) in length. The pipe which contains the utility lines and holds the torch is approximately 2 inches (5.1 cm) in diameter. Electrical requirements for the PLASMOX® torch range from 250-450 volts at current levels from 250-450 amperes.

For the Limited Engineering Scale Tests, the average electrical loads were 418 volts and 342 amperes for an average plasma torch power level of 143 kW. The MGC-designed PLASMOX® torch has its own water-cooling system. It operates under a water pressure of about 100 psi (7 bars) circulating 6.8 gallons (26.6 liters) per minute of cooling water

through the torch. Nitrogen plasma gas is fed into the torch at a gas pressure of about 96 psi (6.5 bars) at a rate of approximately 314 standard cubic feet (8.9 normal cubic meters) per hour.

The electrode is slowly consumed by rotation of the arc around its inner surface. The rotation is the result of an MGC-proprietary "vortex generator" in the torch that "swirls" the plasma gas, causing it to spin the arc. Unlike similar torch designs (e.g., Plasma Energy Corporation (PEC), RETECH, Inc.) the plasma gas pressure does not need to be varied to move the arc along the length of the electrode. The PLASMOX<sup>®</sup> electrode is much smaller than comparable torch designs which apparently precludes this requirement. The elimination of the need to vary the plasma gas pressure also helps to simplify the operation of the torch and reduces one of the factors that could fail and lead to catastrophic torch failure.



Figure 5-4 - PLASMOX® RIF-2 Plasma Torch

# **5.2.2** PLASMOX<sup>®</sup> Electrode Life

The copper electrode for the PLASMOX® torch is about half the size of the electrodes of plasma torches of comparable designs. For example, the PLASMOX® electrode is about 0.75 inches (1.9 cm) in diameter and 4 inches (10.2 cm) long. This contrasts with the similar rear electrodes from other suppliers which are about 1.25 inches (3.2 cm) in diameter and 8.5 inches (21.6 cm) long. This significant reduction in electrode size is a singularly unique characteristic of the MGC-designed PLASMOX® torch. The literature is generally in agreement that electrode life is proportional to the amount of consumable material in the electrode. The PLASMOX® electrode design runs counter to this proposition.

This smaller electrode provides a significant advantage to the PLASMOX<sup>®</sup> process by reducing the volume of water required to cool the electrode. For example comparable torches from other torch suppliers require a cooling water quantity of about 30 gpm at a pressure of over 200 psi; the PLASMOX<sup>®</sup> torch requires cooling water at 6.8 gpm at a pressure of 100 psi. This significant reduction in cooling water results in a much more efficient, economical and safe operating environment. The reduced volume of water also minimizes the risk of over-pressurization of the plasma reactor in the event that a catastrophic torch failure occurs.

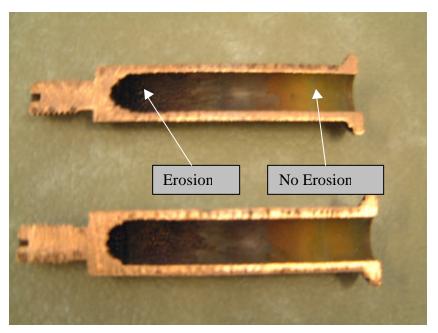


Figure 5-4 - PLASMOX® Electrode

MGC Plasma operators routinely replace the PLASMOX<sup>®</sup> electrode after a maximum of 20 hours of operation. During the final test run (HD-3/4) the electrode was operated for 18 hours and 40 minutes. Inspection of the electrode (see Figure 5-4<sup>21</sup>) showed very little erosion or wear. According to the Burns and Roe/MGC, this erosion is typical of PLASMOX<sup>®</sup> electrode wear patterns. Based on these observations, it appears that the

PLASMOX<sup>®</sup> electrode is highly reliable, with a life that will readily meet the PLASMOX<sup>®</sup> 20 hour standards. With additional testing it may be possible to upgrade the PLASMOX<sup>®</sup> electrode life standards significantly beyond 20 hours.

#### **5.2.3** Torch Failures

A steam excursion can occur if an electrode leak due to erosion is large enough to cause a large volume of water to be discharged into a hot plasma processing reactor. Steam excursions are relevant to water-cooled torches. In the case of the PLASMOX® torch, a steam excursion occurred on 10 January during the Work-Up Test Run. As discussed in Section 4.2.1 above, the torched failed during this test causing 10.5 gallons (40 liters) of cooling water to be released into the vessel. This liquid immediately turned to steam. The system automatically reacted to this pressure spike by increasing the fan speed to restore the negative pressure in the system. The reactor vessel readily contained this pressure spike. The plasma torch was removed within 5 minutes of the event and the vessel resealed. Upon inspection of the failed electrode it was concluded that excessive erosion in a concentrated area of the electrode caused the problem. The liquid feed system was suspected to have forced solid material into the electrode, forming an area of high electrical resistance and erosion. The feed system was modified by eliminating aspirating air and this alleviated the problem during the subsequent test runs.

The reason there was no overpressure failure in the plasma reactor was probably due to the small volume of water (10.5 gallons) which was released into the reactor vessel. Because of the small electrode the entire water cooling system contains only about 80 gallons (300 liters) of circulating water. This relatively low quantity of cooling water passing through the torch is much less than comparable torches (e.g., 6.8 gpm vs 30 gpm). Because of this difference the PLASMOX<sup>®</sup> torch is less susceptible to catastrophic steam excursions/over-pressurization and inherently safer than other comparable plasma torches.

The RIF-2 system may have been able to fully respond to the steam surge generated by the release of cooling water into the plasma reactor if the Off-Gas System piping after the quench had been sized to accommodate such an event. Because of a pinch point between the direct quench and first scrubber, the Off-Gas System fan pulled a significant negative pressure on the first scrubber, causing some deformation of the Scrubber 1 wall.

Burns and Roe/MGC has incorporated steam excursion response into its commercial-scale systems. On the RIF-8 unit which Stone & Webster inspected during the Limited Engineering Scale Tests, two large expansion tanks under slight vacuum are connected to the plasma reactor vessel via two rupture disks. If the pressure in the plasma reactor increased due to a steam excursion, the excess steam would be vented into the two expansion tanks. This design is incorporated in the MGC plasma units at Münster and ZWILAG. A similar design could be used for an NSCMP application.

#### 5.3 Rapid Oxidation Chamber and Off-Gas Treatment

As discussed in Section 3, the ROC was designed and fabricated to incinerate picric acid fed directly into the chamber. Therefore, control of the unit was independent of the rest of the

RIF-2 Unit. Similarly, the off-gas treatment system consisting of the Rapid Quench, scrubbers and exhaust fan were designed to treat the effluent from the batch processing of solid and liquid wastes, not continuous processing of liquid organic wastes as in the Limited Engineering Scale tests. Because of these mismatches, the operation of the these RIF-2 systems was not as efficient as might have been if equipment had been designed for the expected conditions. The inefficiencies of the RIF-2 subsystems when treating liquid organic wastes are discussed below.

### **5.3.1** Rapid Oxidation Chamber

Burns and Roe/MGC were concerned about controlling the temperature of the refractory lining in the ROC during all of the Limited Engineering Scale tests because of the need to have a working unit available for the Albanian campaign. The ROC temperature was controlled by adjusting one or more of the following: the propane fuel to the ROC; oxygen flow to the ROC; and air flow to the burner. Reaction gases exited the plasma reactor at between 850°C and 900°C, while the top of the ROC was operated between 820°C and 890°C. A separate controller maintained the mid-point of the ROC between 1030°C and 1060°C by adjusting the amount of air and propane to the ROC burner; oxygen flow to the ROC was adjusted manually by the operator.

It appears that the process controller on the ROC was not adequately programmed for responding to the energy in the plasma reactor gas. As a result, the heating value of the product gas from the plasma reactor limited the amount of product gas that could be fed to the ROC, which effected the rate of liquid feed that could be processed.

Burns and Roe/MGC based the proposed neutralent feed rates on the anticipated heating values of the product gas. Because of the higher organic content, and subsequently higher thermal content, the feed rate of the HD in MEA neutralent simulant was half the feed rate of the GB in MEA neutralent simulant. Burns and Roe/MGC also used air in the ROC, in addition to oxygen, in order to attenuate the temperature in the ROC while achieving the same level of oxidation. No air was used during the GB test runs.

It is reasonable to assume that the design of the RIF-2 ROC for destruction of directly injected Picric Acid limited the amount of simulant neutralent that could be processed in the RIF-2 reactor. Stone & Webster believes that an ROC designed specifically for treatment of plasma reactor gases, and with process control designed to respond to the heating value of the plasma reactor gases, should be capable of processing higher waste feed rates than were demonstrated during the tests.

# **5.3.2** Off-Gas Treatment System

The Off-Gas System performed as designed during the Limited Engineering Scale tests. With the exception of the water release into the plasma reactor and subsequent steam excursion, there were no upsets in the operation. During the steam excursion incident, the RIF-2 system blower responded to the increase in pressure caused by the steam by pulling more gas through the Off-Gas System and ROC. The diameter of the pipe after the Rapid

Quench restricted the flow of gas such that the system blower pulled a vacuum on the first scrubber vessel. The heat and vacuum caused the vessel to warp.

According to Burns and Roe/MGC the Off-Gas Treatment System on the RIF-2 Unit was not designed to handle steam excursions as experienced during the Work-Up run. MGC reviewed the hydraulics for all sections of the Off-Gas Treatment System in order to confirm that it met operating requirements for the Albanian Campaign.

An Off-Gas Treatment System can be designed to safely treat the off-gas generated from the treatment of NSCMP waste streams. This subsystem could also be designed to accommodate a steam excursion. None of the conditions in the RIF-2 Off-Gas system require unique engineering practices.

### 5.4 Standard Operating Procedures, Training and Safety

MGC has several highly trained, experienced individuals who were well qualified to operate and maintain the PLASMOX® plasma heating systems. Many of these personnel have excellent academic and scientific backgrounds from which to become highly proficient in developing the proper procedures and instructional manuals. In addition, they would be highly qualified to conduct training programs for personnel involved in the operations and maintenance of MGC plasma processing systems. However, it appears that no formal documents have been prepared which encompass operating procedures, maintenance or training.

MGC has incorporated several engineered safety systems into the RIF-2 and RIF-8 units. These include emergency power supply and low cooling water volume in the plasma torch. In other areas, Stone & Webster observed the need for additional safety measures. These operational and safety issues are discussed below.

### **5.4.1** Operational and Emergency Procedures Manual

To our knowledge, Burns and Roe/MGC has no operational or emergency procedures manual for the RIF-2 PLASMOX® system. All training in operational procedures is conducted through "On the Job Training (OJT)". One MGC worker reported that Procedures Manuals were being prepared for the RIF-8 units (1.2 MW) for the operations which are underway in Zwilag, Switzerland and Munster, Germany. Although this report was not verified, Stone & Webster did note that SOPs were developed and available for the RIF-8 Unit located at the Muttenz facility. Burns and Roe has stated that they would be responsible for developing and promulgating operational and emergency procedures manuals for any MGC plasma system selected for waste processing in the U.S.

#### **5.4.2** Training Program

MGC has no specific organized program to train plasma system operators or technicians. Training consists primarily of "On the Job Training (OJT)" of personnel. One MGC employee stated that on a jobsite the trained MGC manager often has workers assigned to him that are completely inexperienced in plasma operations. Burns and Roe has stated that Burns and Roe would be responsible for developing training materials and conducting all

training related to the operation and maintenance of any MGC plasma system selected for waste processing in the United States.

### **5.4.3** Emergency Power Supply

An emergency generator must be readily available to rapidly withdraw the plasma torch from the reactor in the event of failure of external power. Otherwise significant damage to the plasma torch could result along with the possibility of a steam excursion. Burns and Roe/MGC assured Stone & Webster that an emergency generator was on standby at their Müttenz facility to take over in the event of a failure in the external power supply.

An uninterruptible power supply (UPS) would eliminate any delays in bringing an emergency power supply on-line. There is a possibility that the integrity of the plasma torch could be compromised in the short time it would take to start up an emergency generator. Although it is a relatively expensive power system, a UPS would provide greater assurance that the destruction process would not be jeopardized by a minor occurrence such as a thunderstorm. In addition, it would reduce the number of emergency procedures to be implemented, and thereby reduce the potential worker and system mishaps that are inherent in these types of unanticipated actions. The use of a UPS on NSCMP applications should be investigated as an additional safety measure.

### **5.4.4** Engineered Safety Designs

As discussed previously, the major process safety concern associated with water-cooled plasma torch use involves coolant water leaks. This event can result in a steam excursion and the release of dangerous live steam and hazardous materials into the work area. There are two engineered approaches to minimizing the risk of a torch failure: Reduction in volume of water that could enter the reactor; and use of expansion tanks that could accommodate the volume of steam generated. The PLASMOX® plasma systems have the capability to readily incorporate both of these engineered safety features.

Compared to torches of equivalent size and power rating, the PLASMOX<sup>®</sup> torch design significantly reduces the volume of coolant water through the torch. The PLASMOX<sup>®</sup> system can achieve this because of the small mass of the electrode used, compared to torches of similar power raters. The PLASMOX<sup>®</sup> design favorably distinguishes it from other water-cooled electrode plasma torches.

A smaller volume of cooling water does not eliminate the possibility of a steam excursion. Therefore, a plasma system treating hazardous wastes, especially NSCMP waste, should be capable of rapidly and safely respond to steam excursions. The pressure relief system on the RIF-8 Unit, discussed previously, is an example of a reliable approach to responding to the steam excursions that are probable when using water-cooled torches.

#### 5.4.5 Worker Safety

The RIF-2 Unit that was used for the Limited Engineering Scale Testing of the PLASMOX<sup>®</sup> technology was designed for experimental and technology demonstration/development use. As such it did not incorporate many of the worker safety features that would be considered normal for commercial applications. For example, only warning tape strung between two

railings separated workers from high voltage cables leading to the torch and the high temperature plasma reactor lid. A commercial unit will need to include physical barriers to prevent workers from coming in contact with electrical hazards and high temperature surfaces.

### 5.4.6 Availability of Plasma Systems

After completing the Limited Engineering Scale Testing of the PLASMOX® technology, Stone & Webster undertook a survey of available plasma technologies that would potentially be available for implementation at a NSCMP fixed facility to treat NSCMP wastes. The survey identified 15 potentially viable suppliers of plasma systems. Of the 15 technology supplies, five, in addition to Burns and Roe/MGC were visited in order to asses the viability of these systems.

The preliminary conclusion of this survey is that several commercial alternatives to the PLASMOX® plasma system exist. These systems use a variety of torches, including non-transferred and graphite torch designs, that offer advantages and disadvantages compared to the PLASMOX® torch system. The availability of competing systems provides a competitive commercial environment for plasma technologies that is not present for some of the other competing technologies for the destruction of NSCMP wastes.

#### 6.0 EFFLUENT CHARACTERIZATION

Three categories of effluent streams were sampled and analyzed during the Limited Engineering Scale Tests:

- 1) Solid and liquid grab samples from the completion of all test runs plus liquid grab samples from the beginning of all test runs;
- 2) Continuous on-line analysis of gas samples during all test runs; and
- 3) Grab samples from specific product and stack gas streams during portions of all test runs.

All samples were collected by TRC, with the exception of samples collected by EAI Corporation for analysis of agent and agent by-products during the GB Rinsate and RRS Red Neutralent test runs. All samples collected by TRC were submitted to independent laboratories for analysis. Appendix A includes the full report by TRC on the sampling and analysis activities. Appendix B includes the EAI analyses.

The report by TRC provides descriptions of the sample collection and analytical methods for all analyses. Analyses were completed by Phillips Analytical and SWRI.

This section summarizes the results of the analyses of effluent streams from the GB Rinsate, HD in MEA Simulant, simulated CAIS, DF simulant and RRS Red Neutralent test runs.

### **6.1 Solid Sample Analyses**

The Sampling and Analysis Plan (SAP) specified collection and analysis of solid residue from the TRBP (Location 4), solid residue on Scrubber Filters (Location 5) and solid residue from reactor (Location 7). If sufficient quantities of solids from the TRBP and reactor were available, the SAP specified that the samples be analyzed for TCLP metals, SVOCs, VOCs and RCRA characteristics. The filter solids were to be analyzed for metals in order to assess corrosion degradation products.

Sufficient quantities of solids for a full TCLP analysis were collected only for the Reactor Residues (Location 7) from the GB Rinsate Test Run (Run 1) and the Reactor Residues from the RRS Red Neutralent Test Run (Run 9). In the cases where insufficient quantities of solid residue were available to complete the full TCLP, the solids were analyzed for metals. Volatile TCLP analyses were completed for the reactor residues collected after all of the test runs. Semivolatile TCLP analysis was completed for the reactor residue collected after the third DF Simulant Test Run (Run 8). Insufficient quantities of solid residue from the TRBPs were available to complete any TCLP analyses.

Attempts were made to collect solid residues from the TRBP after each test run. However, it most cases, there was very little residue available from the TRBPs. Solid residue from the reactor was collected at the completion of each feed campaign, with the exception of the HD in MEA Simulant campaign, where the system was shut down and the reactor opened after the second HD in MEA Test Run (Run 3). Therefore, there are reactor residue samples from Test Runs 1, 3, 5, 8 and 9. The reactor residue samples collected varied depending on the feed stream. In most cases the material consisted of flakes or powder that collected in the catch plate at the base of the reactor.

Scrubber A filters from all of the test runs were collected after completion of the test run and submitted for metals analysis. Scrubber B filters were also collected after the completion of each campaign (Test Runs 1, 4, 5, 8 and 9). The scrubber filters were analyzed for metals. Scrubber A and B filters were composited for analysis, except for the filters collected after the RRS Red Neutralent test run (Run 9) where both filters were analyzed separately.

#### 6.1.1 TRBP Solids

Solid samples were collected for analysis after each of the validation test runs, except for the last HD Neutralent Simulant run (Run 4) and the first DF Simulant run (Run 6). There was insufficient mass of material available to conduct a full TCLP analysis. Therefore, the solids were analyzed for metals only. The results of the metal analyses of the TRBP solids are shown in **Table 6-1**. Solid material in the TRBPs at the conclusion of the third DF Simulant test run (Run 8) and the RRS Red Neutralent test run (Run 9) were analyzed for bulk metals. The other TRBP solid samples (from Runs 1, 2, 3, 5 and 7) were analyzed for RCRA-8 metals.

The TCLP limits for the RCRA metals are shown in the second column of **Table 6-1**.

The normal operating procedure for shutting down the PLASMOX® system includes thorough melting of the slag in the reactor after liquid feed has been stopped, followed by pouring of the slag into the collection pan. Test Run GB-2 was stopped while feed was still being introduced into the plasma reactor. Because the test run was aborted: organic feed material flowed onto the slag while the crucible was not rotating; the slag in the reactor was not thoroughly melted and mixed; and the melt was not poured into the pan.

The slag from Test Run GB-2 was chiseled out of the crucible after the reactor had cooled. It is possible that the top layer of this cooled slag contained partially-processed organophosphorus feed material. The high TOC and phosphorus content of the samples from this slag may have resulted from this situation and the fact that the slag sample from Test GB-2 was not as thoroughly mixed and homogeneous as samples from the other runs.

The concentration of silicon in the slag from Test Run HD-3/4 is noticeably lower than the silicon content of the slag from the other five test runs. Similarly, the concentrations of most of the other compounds, including metals, are higher as compared to the other samples. The major difference between Test Run HD-3/4 and the other test runs is that it was conducted for 12 hours, whereas the other tests were only run for six hours. The data in Table 4-4 show that of the 22 kg of initial slag charge for Test Run HD-3/4, only 18 kg of slag, approximately 81%, remained. This compares with the other data in Tables 4-3 and 4-4 that show that in all of the other test runs more than 90% of the initial charge was recovered.

The data indicate that the slag charge that is volatilized and driven off in the plasma reactor contains a higher concentration of silicon than is in the melt. If the concentration were equivalent to the concentration in the melt then there would be no appreciable difference in the make-up of the melt between the six-hour runs and the one 12-hour run.

A higher silicon concentration in the gaseous phase could cause plugging in downstream systems depending on where, and if, the silicon deposits. Although the experience of other

plasma torch operators that use glass as a slag material do not support the theory that silicon plates out on system internals, <sup>22</sup> the test data from the Limited Engineering Scale Tests indicate that this is a possibility. Analysis of the solid material collected from the reactor after Test Run HD-1 indicate that silicon does collect in solids in the system piping. As shown in Table 6-2, a portion of the material collected from the reactor outlet piping contained 33% silicon. The composition of more than 90% of the particulate material collected in the filters during Test Runs GB-1 and HD-2 is unknown. It is likely that this material contains slag charge that was volatilized and entrained through the system. Additional testing of plasma systems should include an analysis of how this material is formed and deposited and of the extent of its deposition in the system piping. Of primary interest is whether this solid material is unique to the NSCMP feeds tested, the PLASMOX® system or the use of glass as the melt material.

### **6.1.2** Trace Metals in the Slag

Analysis of trace metals in the slag material show a decrease in the concentrations of chromium, iron and copper between the GB and HD neutralent simulant test runs. The most appreciable decrease occurs for chromium. None of these compounds were present in the simulant feeds tested. Chromium is not expected to be present at appreciable concentrations in the glass and limestone slag charge. However, the new refractory material in the PLASMOX<sup>®</sup> crucible used for these test runs contained 30% Cr<sub>2</sub>O<sub>3</sub>.<sup>23</sup>

The decrease in metal concentrations, especially chromium, in the slag is likely attributable to the conditioning of the new crucible used for these tests. This assumption is supported by the large decrease in chromium concentration in the slag and in the stack gas (Table 6-8) as the tests progressed. The chromium content in the slag after Test Run GB-1 was 2,210 mg/kg. Four test runs later, after Test Run HD-2, the chromium content in the slag had been reduced by an order of magnitude to 94.8 mg/kg. The chromium concentration in the stack gas during Test Run GB-1 was about 1 mg/m<sup>3</sup>, while the concentrations of copper, iron and zinc were all about 0.1 mg/m<sup>3</sup> each (see Table 6-8). The HD-2 stack gas contains a lower concentration of chromium (0.36 mg/m<sup>3</sup>) and higher concentrations of the other trace metals (0.14 to 2.9 mg/m<sup>3</sup>). However, the stack gas data account for only a small percentage of refractory material. As shown in Table 6-8, less than 1% of the particulate material is accounted for in the analysis. If this data were fully consistent with the breaking-in of new refractory material, then one would expect a higher percentage of refractory material to be represented in the particulate analysis. According to Burns and Roe/MGC, emissions of chromium decrease over time as the refractory material is conditioned.<sup>24</sup> Similarly, it would be expected that other refractory material would decrease. Stone and Webster has asked Burns and Roe/MGC to confirm this assumption and to provide information on the composition of the remaining 99% of stack gas particulate. As discussed above in Section 6.1.1, it is likely that the majority of the particulate material originates from the slag.

# 6.1.3 Leaching Properties of Slag

Table 6-1 provides the results of leaching calculations for several metals and TOC. Only one metal detected, chromium, is included in the EPA's list of toxic compounds in 40 CFR

261.24. Zinc is also regulated under Universal Treatment Standards (UTS) for disposal of wastes generated from the treatment of hazardous wastes. These analyses were conducted using Swiss methods, with the understanding that these methods were consistent with US EPA requirements. The analytical results report the concentration of only one of the eight RCRA metals (chromium). Stone & Webster has requested that Burns and Roe confirm whether the other seven RCRA metals (arsenic, barium, cadmium, lead, mercury, selenium and silver) were included in the analysis and not detected, or whether they were never analyzed. Barium was not reported in the leaching results but was included in the gross analysis of the slag and reported as not detected at the detection limit.

The Swiss methods calculate a mean value based on 24-hour and 48-hour leaching tests. However, even if the values from the two leaching periods were added, instead of averaged, the resulting values for chromium would be well below the Maximum Concentration standards for the EPA Toxicity Characteristic (5 mg/L). The concentrations of zinc are also below the UTS. Therefore, based on the data from the Limited Engineering Scale Tests, the slag material could be disposed in a non-RCRA landfill, assuming that chromium was the only RCRA 8 metal detected. As discussed above, the likely source of the chromium is the refractory material used in the crucible. Stone & Webster has requested that Burns and Roe provide the composition of the refractory material. If the refractory does not provide a source for any of the other RCRA metals, then the conclusion concerning the disposability of the slag is still valid. In order to confirm this, Stone & Webster has submitted a sample of the slag from Test Run GB-3 for analysis, including a full RCRA TCLP analysis.

### 6.1.4 Analysis of Reactor Exit Piping Solid Material

Greenish white solid material was found in the exit piping from the plasma reactor after both Test Run GB-2 and HD-1. Figure 5-3 shows the material in the reactor exhaust piping after Test Run HD-1. The material was predominantly green and appeared to block more than 50% of the cross-sectional area of the piping at the reactor outlet. A sample of the material from Test Run HD-1 was collected and submitted to the laboratory for analysis.

Table 6-2 provides the results of the surface chemical analysis of this material using x-ray diffraction. The analysis is qualitative and not quantitative and provides information only about what elements are present, but not the relative concentrations of elements. The material was non-homogeneous and consisted of three distinct materials: dark green material; light green material and a white material. As the data show, all three composite materials contain chlorine. The second most common element in the white material was silicon. In the dark green material the second most common element was phosphorus. The light green material is predominantly chlorine.

**Table 6-1 – Surface Analysis of Reactor Exit Piping Solid Material** 

	Dark Green Material	<b>Light Green Material</b>	White Material
Element	Relative Portion of	Relative Portion of	Relative Portion of
	Material [%]	Material [%]	Material [%]

Table 6-1 – Surface Analysis of Reactor Exit Piping Solid Material

	Dark Green Material	Light Green Material	White Material
Element	Relative Portion of Material [%]	Relative Portion of Material [%]	Relative Portion of Material [%]
С	0.0%	0.2%	0.1%
О	8.6%	0.4%	5.5%
Na	8.3%	5.8%	4.9%
Al	3.6%	-	0.2%
Si	8.0%	0.8%	33.2%
P	29.9%	-	2.8%
Cl	34.3%	90.7%	49.6%
K	4.8%	2.2%	3.3%
Ca	1.6%	-	0.4%
Cr	0.9%	-	-
Mn	0.6%	-	-

As the sample was very inhomogeneous, 3 spectra at 3 different spots (coloration) were taken.

This data support the theory that slag material is volatilized and potentially deposited in system piping. Additional plasma system testing should focus on the extent and impact of this process. In addition, the data provide some indication of where the phosphorus in the feed material could be accumulating. To further assess whether phosphorus collects on system piping, a sample of the material has been sent to a U.S. analytical laboratory for a complete chemical analysis.

#### **6.2 Liquid Sample Analyses**

Grab samples were collected from Scrubber 1 and Scrubber 2 before and after each GB and HD test. The Test Plan also specified collection and analysis of liquid from the Rapid Quench system. According to Burns and Roe/MGC, the Rapid Quench water was combined with the Scrubber 1 water.

Liquid samples from the GB test runs were analyzed for aldehydes, TOC, total cyanide, phosphate, nitrate, MEA( $C_2H_7NO$ ), DMMP( $C_3H_9O_3P$ ), SVOCs and VOCs. Liquid samples from the HD test runs were analyzed for aldehydes, TOC, chloride, total cyanide, dioxinsfurans, nitrate, MEA( $C_2H_7NO$ ), SVOCs and VOCs. Results of the liquid sample analyses are presented in Tables 6-3 and 6-4.

This document was prepared under contract with the United States Army for the sole purpose of evaluating the identified technology for potential application in the United States Army Chemical Demilitarization Program (CDP), based on information available to the reviewer at the time of the evaluation. Any opinions, findings, recommendations or conclusions expressed are stated in the context of the particular considerations of the CDP, and are not intended for use or reference in any way by any other party for any other purpose.

Stone & Webster also requested that a sample of condensate from the stack particulate sampling train, collected during Test Run GB-1, be submitted for analysis. This liquid was pinkish in color. According to Burns and Roe, this sample contained 25.7 grams of phosphorus per kilogram of liquid. This analysis is not consistent with other available data, since it corresponds to approximately 10 kg of phosphorus in the stack gas during the six hour test run. Only 1.5 kg of phosphorus were fed into the system over that time period. Stone & Webster has requested further confirmation of this analysis in order to complete a phosphorus balance.

**Table 6-2 Scrubber Water Analyses – GB Test Runs** 

			Scrubber	· 1/Quench	Scrub	ober 2	Scrubber 1	Scrubber 2	Scrubber 1	Scrubber 2
Parameter	Unit	Method	Before Test GB-1 (Baseline)	After Test GB-1	Before Test GB-1 (Baseline)	After Test GB-1	After Test GB-2	After Test GB-2	After Test GB-3	After Test GB-3
Chloride	[mg/l]	VS-124 (IC)	20.5	81.1	<0.08	5.83	120.5	10.0	194.6	13.1
Cyanide	[mg/l]	VS-143	< 0.004	< 0.004	0.008	0.02	0.014	0.034	0.018	0.036
Nitrate	[mg/l]	VS-124 (IC)	4.21	58.8	<0.36	24.5	102	57.6	184	91.0
Phosphorus	[mgP/l]	VS-141	0.05	34.5	7.00	25.5	60.5	61.5	140	120
Formaldehyde	[mg/l]	EPA 8315A (HPLC)	0.838	0.167	0.050	0.158	1.842	0.175	0.097	0.118
Acetaldehyde	[mg/l]	EPA 8315A (HPLC)	0.053	< 0.03	0.063	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
TOC	[mg/l]	VS-118	10.7	11.9	17.8	18.1	16.5	30.0	20.1	27.2
PAH**	[mg/l]	VS-103b	0.31	0.69	0.21	0.33	0.47	0.22	0.36	0.68
MEA	[mg/l]	NIOSH 2007	<5	<5	<5	<5	<5	<5	<5	<5
DMMP	[mg/l]	(*)	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5

#### **NOTES**

Scrubber water was not changed between test runs.

<sup>\* :</sup> this method is not included in Dr. Meyer AG accreditation program. Stone & Webster has requested information regarding what method was used.

<sup>\*\* :</sup> sum of 16 compounds, detailed results see Appendix A for PAH I+II

Table 6-3 - Scrubber Water Analyses - HD Test Runs

			Scrubber 1	/Quench	Scrubber 2		Scrubber 1	Scrubber 2	Scrubber 1	Scrubber 2	Scrubber 1	Scrubber 2
Parameter	Unit	Method	Before Test HD-1 (Baseline)	After Test HD-1	Before Test HD-1 (Baseline)	After Test HD-1	After Test HD-2	After Test HD-2	After Test HD-3	After Test HD-3	After Test HD-4	After Test HD-4
Chloride	[mg/l]	VS-124 (IC)	10.1	2560	8.44	260	2720	578	929	1440	525	1999
Cyanide	[mg/l]	VS-143	< 0.004	0.008	0.014	0.008	0.01	0.024	< 0.004	0.008	0.006	< 0.004
Nitrate	[mg/l]	VS-124 (IC)	13.8	23.7	8.96	11.1	59.2	12.1	57.7	18.7	60.9	65.8
Formaldehyde	[mg/l]	EPA 8315A (HPLC)	0.547	0.675	0.104	0.302	0.350	0.064	0.196	0.099	0.165	0.067
Acetaldehyde	[mg/l]	EPA 8315A (HPLC)	< 0.03	0.034	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03
TOC	[mg/l]	VS-118	5.0	4.1	21.8	17.7	3.0	20.8	2.0	26.6	1.8	28.8
PAH**	[mg/l]	VS-103b	0.48	0.37	0.18	0.19	< 16	0.26	< 16	< 16	< 16	< 16
MEA	[mg/l]	NIOSH 2007	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
Dioxins Furans***	[ng/l TE]	VS-122	9.65 - 9.74	10.95 - 11.11	0.21 - 0.56	0.15 - 1.50	2.98 - 3.06	0.05 - 0.44	1.88 - 1.98	0.00 - 1.23	1.21 - 1.39	0.01 - 0.50

#### **NOTES**

- \* : this method is not included in Dr Meyer AG accreditation program
- \*\* : sum of 16 compounds, detailed results see Appendix A for PAH I+II

Fresh scrubber water added before Test Run HD-1. Scrubber water was not changed between test runs.

<sup>\*\*\* :</sup> sum of 2,3,7,8-chlorine substituted congeners of dioxins and furans, evaluation according to the International Toxicity Equivalent Factors (ITEF); results reported as max and min Toxic Equivalents (TE) – max TE value reports concentration at the detection limit for compounds not detected; detailed results see Appendix A for dioxins/furans 1-10

The data in Tables 6-3 and 6-4 are presented for water collected from Scrubber 1 and Scrubber 2. Scrubber 1 water and the water from the Rapid Quench were hydraulically connected and, therefore, the Scrubber 1 water results reported in Tables 6-3 and 6-4 represent the combined results from Scrubber 1 and the Rapid Quench. To evaluate the results of the tests, nominal values of 600 liters and 400 liters were assumed for the volume of Scrubber 1/Rapid Quench water and Scrubber 2 water, respectively. Actual liquid volumes used in the test were not measured, although it would be expected that water volumes would increase during the tests due to condensation.

Samples were collected from the scrubbers before and after each test run. Note that the analysis of the scrubber water "Before" each of the test runs, with the exception of Test Run 1 is always the analysis "After" the previous test run. For example, the analysis of the Scrubber 2 water before Test Run GB-3 is reported in the "After Test GB-2" column. The Scrubber Water was not changed between the test runs, but was only changed between the GB and HD test run campaigns, i.e. after Test Run GB-3. Complete analyses of the scrubber water samples are included in Appendix A.

#### **6.2.1 Phosphorus Balance**

One of the objectives of the Limited Engineering Scale tests of the PLASMOX<sup>®</sup> system was to determine the fate of phosphorus in the system. To accomplish this objective, the Test Plan required collection of sufficient data to complete a phosphorus material balance. Samples were collected from the slag, scrubber liquid and stack gas and analyzed for phosphorus and phosphorus-containing compounds. The Test Plan specified sampling of the stack gas from Test Run GB-1 and analyzing for phosphorus. The stack gas sample from Test Run GB-1 was not analyzed for phosphorus. Instead, the analytical subcontractor substituted analysis of the stack gas from Test Run HD-2. Because the simulant used in Test Run HD-2 contained no phosphorus, the data is inconclusive. No phosphorus balance can be calculated with the data provided.

An attempt is being made to use the analysis of the pink condensate collected from the particulate sampling train during Test Run GB-1 to calculate a phosphorus balance. Initial estimates

The Pine Bluff Arsenal (PBA) NPDES permit places maximum daily and monthly discharge quantities on phosphate. The maximum daily discharge of phosphate per the PBA permit is 21.8 pounds of phosphorus. The maximum concentration of phosphorus in the scrubber water during the GB test runs was 140 mg/L. This was the maximum concentration after processing 230 liters of GB neutralent simulant over 18 hours of total processing time.

As discussed below in Section 6.2.4, overall test data indicate that the RIF-2 Off-Gas Treatment System does not effectively remove compounds of concern from the ROC exhaust gas. Future modifications would be required for a PLASMOX system designed to treat NSCMP wastes. These modifications will have to include a more effective Off-Gas Treatment System, that will likely result in higher recovery of phosphorus in the scrubbers, with potential impacts on effluent treatment.

#### **6.2.2** Dioxins-Furans in Scrubber Water

Dioxins and furans were detected in all scrubber water samples collected during the HD test runs, including the "clean" scrubber water samples from Scrubber 1 and Scrubber 2 prior to beginning Test Run HD-1. The levels ranged from a high of 11 ng/l to a low of 0.05 ng/l, as shown in Table 6-4. The high values in Table 6-4 were estimated by reporting the concentration of dioxins/furans at the reporting limit for non-detects.

The data from the analysis of the scrubber water before Test Run HD-1 indicate that the scrubbers were contaminated previously, possibly during earlier test campaigns. Burns and Roe/MGC reported that they have witnessed dioxins and furans "leaching" out of previously contaminated systems and contaminating "cleaned" systems. According to Burns and Roe/MGC, "past experience with scrubber systems indicate that even 'cleaned' systems, which were previously contaminated with dioxins and furans, will 'leach' dioxins and furans for as long as 6 months after cleaning and operating." Stone & Webster has requested that Burns and Roe/MGC provide information on previous RIF-2 test campaigns where the scrubbers may have become contaminated.

This assumption is supported by the data. The dioxins/furans data do not show an accumulation trend from test to test as one would expect if dioxins/furans were being generated in the process. For comparison, the concentration of nitrates in the scrubber solutions increases in both scrubbers from Test Run HD-1 to HD-3/4, indicating that nitrates are produced in the process and accumulate in the scrubbers. The maximum concentrations of dioxins/furans are recorded after Test Run HD-1 in both Scrubber 1 and Scrubber 2. Concentrations decrease over time, most likely indicating that residue material has been leached out and dissipates over time. This leaching out is therefore a possible source of dioxins/furans detected in the stack gas.

### 6.2.3 Total Organic Carbon, Chloride, Cyanide and Nitrates in Scrubber Water

A primary evaluation criteria for determining the viability of disposing of scrubber water generated during operation of the PLASMOX® system was a limit of 25 ppm on Total Organic Carbon (TOC). This value is based on the existing TOC concentration permitted in wastewater discharged from the Pine Bluff Arsenal Central Waste Treatment facility to the Arkansas River. Therefore, it is a conservative evaluation criteria. It is expected that wastewater from a PLASMOX® facility or any neutralent post-treatment technology would be discharged to a wastewater treatment facility before ultimate disposal. The 25 ppm criteria provides a broad evaluation of the viability of disposing of the waste water.

Because the scrubber water was not changed between test runs, one would expect that the concentration of TOC would increase from run to run. In general, this trend is observed in the data in Tables 6-3 and 6-4. In the GB test runs, the concentration of TOC in Scrubber 1 and Rapid Quench water increases from run to run and does not exceed 25 ppm. The concentration of TOC in Scrubber 2 water increases during Test Runs GB-1 and GB-2 and decreases slightly during Test Run GB-3.

The other organic compounds detected in the scrubber water (aldehydes and PAHs) did not appear to accumulate like the TOC. The concentration of aldehydes in both the GB and HD test runs decreased from run to run. Concentrations of PAHs increased slightly during the GB test runs and decreased slightly during the HD runs. Overall the data show little accumulation over the course of both sets of test runs.

In addition to phosphate and TOC, the PBA NPDES permit requires the reporting of the maximum daily and monthly discharge quantities of chloride. The chloride data from the HD test runs indicate that the scrubbers on the RIF-2 system are ineffective in removing acid gases. The quantity of chloride recovered peaks after the first test run, HD-1, and decreases on each subsequent test. During Test Run HD-3/4 the chloride concentration in the combined scrubber liquids decreases, indicating that acid gases were stripped – instead of being scrubbed – from the scrubber liquid during this 12-hour test run. Based on the test data, it is probable that the scrubber effluent would require pretreatment depending on site-specific conations, since the EPA maximum ambient water quality criteria is 860 mg/L and the long term criteria is 230 mg/L.

The current PBA permit does not list nitrates and cyanides, both of which are apparently formed in the process and do accumulate in the scrubber water. However, no definitive conclusions can be drawn from the data as to how a PLASMOX<sup>®</sup> unit might be permitted because of the poor performance of the scrubber system. Cyanide values during testing were below levels typically required under pretreatment standards, and close to EPA ambient water quality criteria (0.022 mg/L maximum, 0.055 mg/L long term). However, under the operating conditions of the RIF-2 system scrubbers, it is likely that HCN, like the chloride, was stripped and that the data represent a 'best-case' scenario. Ultimately phosphate, chloride, nitrate and cyanide discharge levels would depend on site-specific loading in the receiving stream, flow and whole effluent toxicity factors.

#### **6.2.4** Scrubber Performance

As the discussion of acid gas removal above indicates, the overall effectiveness of the scrubber system installed on the RIF-2 PLASMOX<sup>®</sup> unit is not sufficient to adequately treat the exhaust gases from the ROC. This conclusion is supported by the system's performance in removing phosphorus and particulates, in addition to chlorides.

The phosphorus data for the GB test runs in Table 6-3 indicate that the scrubbers are approximately 65% efficient in removing phosphorus. This calculation, shown in Appendix C, assumes that the efficiency of each scrubber is equal.

Similarly, a review of the stack gas data in Table 6-8 shows a very high particulate loading, indicating that the scrubber system is ineffective in removing particulates from the ROC exhaust. Burns and Roe/MGC have claimed that the high concentration of particulate in the stack gas resulted from conditioning of the refractory in the new crucible used for these tests. They have suggested that HEPA filters could be used to reduce the particulate emissions. However, at the rates measured during these tests (up to 90 gm/hr), the particle loading on a typical HEPA filter would be exceeded in 20 hours of operation.

The chloride, phosphorus and particulate data indicate that the RIF-2 PLASMOX® unit scrubber system is ineffective in reducing the concentrations of these compounds in exhaust gases from the ROC and plasma reactor. In a production unit PLASMOX® system designed for treatment of NSCMP wastes, the scrubbers would be designed to meet specific removal efficiencies for compounds of concern. These modifications will likely result in higher recovery of phosphorus, chlorides and particulates in the scrubbers, with potential impacts on effluent treatment. Ultimately, effluent acceptability will be a function of unit throughput as well as location-specific Total Maximum Daily Loading (TMDL) requirements and potential treatment works operating conditions (i.e. removal capabilities and flow).

## **6.3 Gas Sample Analyses**

Stack gases downstream of the system blower were continuously monitored during all six test runs for oxygen, carbon dioxide, carbon monoxide, NO<sub>x</sub> and organic carbon. Graphic displays of this data are presented in Figures 4-1 through 4-6. Averages of the continuous stack gas data for each of the six test runs are summarized in Table 6-6. The volumetric flow of stack gas was measured during Test Run GB-1 and HD-2. The estimated volumetric flow of exhaust gas from the scrubbers is 271 Nm³/hr in Test Run GB-1 and 383 Nm³/hr in Test Run HD-2.

Grab samples from the stack were also collected during Test Runs GB-1, GB-2 and HD-2 and analyzed for specific compounds. The exit gas from the plasma reactor, upstream of the ROC, was analyzed for MEA during Test Runs GB-2 and HD-2. A sample of the gas stream between the ROC and quench was also sampled during Test Run HD-2 and analyzed for dioxins/furans. All stack sampling was conducted by Dr. Graf AG.

During Test Run GB-1 stack gas samples were submitted for analysis of the following compounds: aldehydes; hydrocyanic acid; metals; particulates; monoethanolamine (MEA); dimethyl-methylphosphonate (DMMP); and identification of SVOC's and VOC's (by GC-MS-Fingerprint). The stack gas from Test Run GB-1 was also to be analyzed for phosphorus. This analysis was not completed..

During Test Run HD-2 stack gas samples were submitted for analysis of: particulates; metals; hydrocyanic acid; hydrochloric acid; aldehydes; MEA; dioxins and furans; and identification of SVOC's and VOC's (by GC-MS-Fingerprint). The stack gas from Test Run HD-2 was analyzed for phosphorus and aluminum.

Complete analyses of gas samples are included in Appendix A.

### **6.3.1** Destruction of MEA

It was noted during the tests that the location of the liquid feed injection nozzle into the lid of the plasma reactor was adjacent to the reactor exhaust port. Based on the geometry of the feed and reactor exhaust, it appeared that feed material could be swept out of the reactor without seeing the maximum temperatures and residence time necessary for complete destruction. Current Maximum Achievable Control Technology (MACT) standards for the destruction of hazardous wastes include destruction efficiency standards. To check for the possibility of bypass of feed material and consequential reduction in destruction efficiency,

samples were collected from the transfer line connecting the plasma reactor to the ROC. Samples collected from Test Runs GB-2 and HD-2 at this location were analyzed for MEA.

As shown in Appendix A, <sup>29</sup> the concentration of MEA in both samples was below the analytical detection limits. Based on the available data, an estimate of the plasma reactor destruction efficiency for MEA was calculated. The results are summarized in Table 6-5 below. This estimate is a conservative calculation since it uses the larger stack gas volume instead of the smaller volume of gas which would exit the plasma reactor. A calculation using an estimate of the plasma exhaust gas volume is included in Appendix D and concludes that greater than 99.9999% destruction was achieved.

**%** Test **Total Test** Stack Stack **MEA MEA** in % **Total** Run **Feed MEA** Time **Flow Vol.** (m<sup>3</sup>) Conc. Stack (mg) **Destruction MEA**  $(m^3/hr)$  $(mg/m^3)$ (kg) in Feed (kg) (hrs) 40% 29.52 570\* GB-2 73.8 6.00 3,420 < 0.2>99.9977% <684.0 39.3 HD-2 83% 32.62 6.63 610 4.046 < 0.1<404.6 >99.9988%

**Table 6-4 - Estimated MEA Destruction** 

Notes: \*Flowrate is average flow from Test Run GB-1. No volume flow data available for GB-2.

As shown in Table 6-5, the destruction of MEA in the plasma reactor was greater than 99.99% for both simulant feeds. Proper placement of the liquid feed lance relative to the reactor exhaust should make achieving high destruction efficiencies more reliable.

### 6.3.2 NO<sub>X</sub> Emissions

A primary concern for a plasma system that uses nitrogen as the plasma gas is the production of nitrogen oxides. All three PLASMOX<sup>®</sup> RIF-8 units incorporate Selective Catalytic Reduction (SCR)  $NO_x$  reduction in order to meet environmental standards.

Table 6-6 summarizes the continuous on-line measurement of five key components of the stack gas. NO<sub>x</sub> emission standards applicable to a specific unit will be a function of the unit's location and size as determined in a case-by-case control technology evaluation under Prevention of Significant Deterioration (PSD), Non-attainment New Source Review, or a state minor source construction permit program. Because the PLASMOX system would be subject to the National Emissions Standards for Hazardous Air Pollutants (NESHAP) from Hazardous Waste Combustors (40 CFR 63, Subpart EEE), it is exempt from the New Source Performance Standards (NSPS) for Commercial and Industrial Solid Waste Incinerators (40 CFR Part 60, Subpart CCCC). As a guideline however, the NSPS Subpart CCCC standard for NO<sub>x</sub> emissions could be used as a benchmark for a plasma system. According to the NSPS Subpart CCCC Standards, <sup>30</sup> the maximum emission limit for oxides of nitrogen is 388 ppm by dry volume (@ 7% O<sub>2</sub>). As shown in Table 6-6, the average NO<sub>x</sub> emission value for the three HD test runs was 185 ppm<sub>v</sub> dry volume (ppm<sub>v d</sub>). The maximum NO<sub>x</sub> emissions measured during the tests was during Test Run HD-3/4 where the value was 217 ppm<sub>v,d</sub>. Based only on these limited results it appears that a PLASMOX® system treating NSCMP neutralents will not exceed the maximum NSPS limits for NO<sub>x</sub> emissions.

According to Burns and Roe/MGC,  $^{31}$  three factors effect the degree to which  $NO_x$  is produced in the PLASMOX® system:

- Carbon concentration in the process vessel;
- Nitrogen/oxygen concentration in the vicinity of the plasma arc; and
- PLASMOX<sup>®</sup> system temperature.

**Table 6-5 – Continuous Stack Analyses** 

	O2	CO2	CO *	NOx *	Org. C *
Test Run	%	%	ppm <sub>v,d</sub>	$ppm_{v,d}$	ppm <sub>v,d</sub>
GB-1	$12.5 \pm 0.6$	$6.4 \pm 0.3$	<8	$69 \pm 13$	$3 \pm 0.4$
GB-2	$11.9 \pm 0.6$	$6.5 \pm 0.3$	<8	$136 \pm 16$	$1 \pm 0.5$
GB-3	$10.7 \pm 0.5$	$7.3 \pm 0.4$	<7	$135 \pm 15$	$2 \pm 0.5$
HD-1	$8.6 \pm 0.4$	$8.0 \pm 0.4$	8 ± 6	$160 \pm 17$	$0.5 \pm 0.5$
HD-2	$9.1 \pm 0.5$	$7.7 \pm 0.4$	<6	$178 \pm 20$	$0.5 \pm 0.5$
HD-3/4	$9.9 \pm 0.5$	$7.1 \pm 0.4$	11 ± 6	$217 \pm 24$	$1 \pm 0.4$
Ave. GB-1/3	$11.7 \pm 0.6$	$6.7 \pm 0.3$	<8	113 ± 15	$2 \pm 0.5$
Ave. HD-1/4	$9.2 \pm 0.5$	$7.6 \pm 0.4$	8 ± 6	$185 \pm 20$	$1 \pm 0.5$
Total Ave.	$10.4 \pm 0.5$	$7.2 \pm 0.4$	8 ± 6	149 ± 18	$2 \pm 0.5$

<sup>\*</sup>the concentrations are corrected for the dilution-air effect, converted from  $mg/m^3$  and corrected to 7% oxygen. <sup>32</sup>

The data also indicate that the material being processed influences NO<sub>x</sub> formation.

According to the first factor, because carbon has a higher affinity for oxygen than does nitrogen, increasing the amount of organic material to the plasma reactor will tend to reduce the concentration of  $NO_x$ . Based on this assumption, one would expect that the HD Test Runs to produce less  $NO_x$ , on average, because the organic content in the HD simulant was four times that of the GB simulant. Instead, the  $NO_x$  concentration for the HD test runs was nearly twice that of the GB runs.

According to the second factor cited by Burns and Roe/MGC, the discrepancy could be explained by the higher oxygen content of the HD simulant. The second factor states that the amount of  $NO_x$  produced in the plasma reactor is proportional to the amount of oxygen available.

However, if the primary source of  $NO_x$  were the plasma reactor and  $NO_x$  formation followed the factors outlined by Burns and Roe/MGC, then one would expect Test Run HD-3/4 to have the lowest  $NO_x$  concentration of all six runs. Test Run HD-3/4 combined high organic

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content and low oxygen content because of the simulant processed and the fact that no oxygen was introduced into the plasma reactor during this run. Both of these factors should have resulted in the lowest  $NO_x$  concentration for all six test runs, other factors being equal.

However, all other factors were not equal. Instead, Test Run HD-3/4 had the highest  $NO_x$  concentration, essentially twice the average  $NO_x$  concentration of the GB test runs. As shown in Table 6-7, a major difference between the GB Test Runs and the HD Test Runs was the use of air in the ROC and the consequential increase in oxygen to the system. A second distinguishing feature between the two sets of runs is the difference in composition of the simulants tested. The HD test runs contained chlorine in the feed and reactor exhaust, whereas the GB test runs did not.

No air was fed to the ROC during the GB test runs. However, during all three HD test runs, significant quantities of air (oxygen) were supplied to the ROC. Air was used in the ROC in order to control the temperature by diluting the stream with nitrogen. With the exception of Test Run GB-1, the range of concentrations of  $NO_x$  in the stack gas roughly correlate with the quantity of oxygen fed to the reactor and ROC.

Test Run:	GB-1	GB-2	GB-3	HD-1	HD-2	HD-3/4
O <sub>2</sub> Supply to Reactor Vessel (Nm³/h)	6.4	4.8	4.8 (later in run 2.4)	4.8	5.6	0
O <sub>2</sub> Supply to ROC (Nm³/h)	4.8	4.8	4.8 (later in run 2.4)	3.2 (+ 18.0 air)	18.0 air (later in run 4 O <sub>2</sub> , air off)	35 air + 5.4 O <sub>2</sub>
Total O <sub>2</sub> Supply to System (Nm <sup>3</sup> /h)	11.2	9.6	9.6 (later in run 4.8)	11.8	9.4 (later in run 9.6)	12.7

Table 6-6 - Oxygen Supply to Reactor and ROC

A comparison of the data from Test Run GB-2 and GB-3 with data from Test Run HD-2 also indicates that the composition of the feed material has an impact on  $NO_x$  production. The data do not provide conclusive evidence as to whether the plasma reactor or ROC is the predominant source of  $NO_x$ .

## **6.3.3** Dioxins and Furans

Another environmental concern for high temperature thermal processes treating chlorinated wastes, like the PLASMOX<sup>®</sup> system, is the production of dioxins and furans. Dioxins and furans were detected in both the scrubber water and stack gas during processing of the HD in MEA neutralent simulant. No dioxins/furans were detected above the detection limit in the ROC exit line upstream of the Rapid Quench. <sup>33</sup> As shown in Table 6-8, the dioxins/furans concentration in the stack gas was 0.25 ng-TEQ/m<sup>3</sup>. This is above the NESHAP limit for hazardous waste combustors of 0.20 ng-TEQ/m<sup>3</sup>.

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Burns and Roe/MGC contend that dioxins/furans are not formed in the process because the conditions in the PLASMOX® system are not conducive to their production. Instead, Burns and Roe/MGC have postulated that the presence of dioxins/furans in the stack gas and scrubber water is a result of dioxins and furans that had contaminated the system during previous tests of the RIF-2 unit. According to this theory, the compounds were leached out of the system into the scrubber water and eventually carried-over into the stack gas. As discussed in Section 6.2.2, analysis of the scrubber water support this assumption.

The dioxins/furans concentrations in the stack gas measured during the HD test run are above regulatory limits. However, the likely source of dioxins/furans detected during the test are from previous contamination of the system during other tests. Additional testing of plasma systems should focus on confirming whether dioxins/furans are produced in plasma systems.

#### **6.3.4** Particulates and Metals in Stack Gas

The concentrations of particulates and metals detected in stack gas grab samples are shown in Table 6-8. The particulate concentrations are well above the Maximum Achievable Control Technology (MACT) limit of 34 mg/m³ for hazardous waste combustors. <sup>36</sup> In addition, the chromium concentrations exceed the 97  $\mu$ g/m³ MACT limit. <sup>37</sup> These high concentrations are an indication that the RIF-2 Off-Gas Treatment System is ineffective. These values should be reduced considerably by an improved scrubber and HEPA filter system, which would be standard on any application of the PLASMOX® system for NSCMP wastes.

The HEPA filter was not used during the Limited Engineering Scale tests because Burns and Roe/MGC believed that the wastes being processed would not generate significant particulate matter. As discussed above, the concentration of particulate matter and metals in the stack gas have been attributed to the "breaking in" of the new crucible material. This assumption is based on the fact that, with the exception of phosphorus, none of the metals detected in the stack gas are associated with the feeds processed. Instead, they are components of the crucible refractory material.

Burns and Roe/MGC have provided no historical data that confirm that the concentrations of these metals and particulate levels decrease as the crucible is conditioned, or that crucible materials are ever entrained in the plasma reactor exhaust. What is more likely is that the bulk of the particulate material collected consists of slag material. As discussed in Section 6.1.1, the loss of slag charge material indicates that portions of the melt are volatilized and entrained through the RIF-2 unit. Regardless of the source of the particulate, the use of improved scrubber and HEPA filters will reduce the particulate and metals loading to within regulatory limits. In an NSCMP application, the spent filters could be reprocessed through the plasma system.

**Table 6-7 - Stack Gas Grab Sample Analyses** 

	Concentration	on (mg/m³)	Average Flo	Average Flowrate (g/h)		
	GB-1	HD-2	GB-1	HD-2		
Particulates	$170 \pm 20$	$270 \pm 30$	43 ± 9	$90 \pm 17$		
Chromium	$0.99 \pm 0.20$	$0.36 \pm 0.07$	$0.25 \pm 0.07$	$0.12 \pm 0.03$		
Copper	$0.094 \pm 0.019$	$0.62 \pm 0.13$	$0.024 \pm 0.007$	$0.21 \pm 0.05$		
Iron	$0.090 \pm 0.017$	$0.14 \pm 0.03$	$0.023 \pm 0.006$	$0.05 \pm 0.01$		
Zinc	$0.094 \pm 0.018$	$2.9 \pm 0.6$	$0.024 \pm 0.006$	$1.0 \pm 0.3$		
Phosphorus	-	$2.2 \pm 0.4$	-	$0.8 \pm 0.2$		
Aluminum	-	$0.05 \pm 0.01$	-	0.017 ± 0.004		
Acetaldehyde	$0.41 \pm 0.08$	$0.17 \pm 0.03$	$0.10 \pm 0.03$	$0.06 \pm 0.01$		
Formaldehyde	$0.33 \pm 0.07$	$0.21 \pm 0.04$	$0.08 \pm 0.02$	$0.07 \pm 0.02$		
Sum	$0.74 \pm 0.11$	$0.38 \pm 0.06$	$0.19 \pm 0.04$	$0.13 \pm 0.02$		
HCN	< 1.6	< 2	< 0.4	< 0.6		
HCl	N/A	$0.36 \pm 0.04$	N/A	$0.12 \pm 0.02$		
MEA	< 0.2	< 0.2	< 0.04	< 0.08		
DMMP	< 0.7	N/A	< 0.2	N/A		
Dioxins/Furans*	N/A	$0.25 \pm 0.11$ ng TEQ/m <sup>3</sup>	N/A	0.08 ± 0.03 µg TEQ/h		

<sup>\*</sup> Dioxins/furans concentrations are expressed in Toxicity Equivalents (TEQ), which is the measured concentration times the International Toxicity Equivalence Factor (I-TEF) for each congener.

Additional testing of plasma systems should focus on collection of data to establish the relative contributions of slag and refractory to particulate loadings.

## **6.3.5** Hydrocarbons and Hazardous Air Pollutants

Under the Clean Air Act Amendments of 1990, the U.S. EPA has established standards applicable to emissions of Hazardous Air Pollutants (HAPs). A major HAP source is one which has potential HAP emissions of a single compound greater than 10 tons per year (tpy) or combined HAP emissions greater than 25 tpy. Smaller sources are referred to as area sources. Limits on HAP emissions are established for specific source categories based on Maximum Achievable Control Technology (MACT) based on the characteristics and operations of that category. MACT standards for hazardous waste combustors are established under Subpart EEE that apply to both major and area sources. The MACT

standards require that hazardous waste combustors limit CO concentrations to below 100 ppm<sub>v,d</sub> (@ 7%  $O_2$ ) or the total concentration of hydrocarbons to below 10 ppm<sub>v,d</sub> (@ 7%  $O_2$ ). Compliance with these standards generally limit potential HAP emissions, without establishing additional MACT standards for specific organic HAPs..

The hydrocarbon content of the stack gas was monitored continuously during all six test runs using a Flame Ionization Detector (FID), calibrated with propane. The average hydrocarbon concentration in the stack gas during all six test runs, in ppm<sub>v,d</sub> organic C, is summarized in Table 6-6. The maximum average concentration for CO was 11 ppm<sub>v,d</sub> for Test Run HD-3/4 (see Table 6-6). This is approximately 10% of the permissible limit. Likewise the maximum average hydrocarbon concentration in the stack gas occurred during Test Run GB-1. As shown in Table 6-6, the average hydrocarbon concentration was 3 ppm<sub>v,d</sub>, less than half of the MACT limit. Comparison of the test results against the MACT standards indicates that no post-treatment will be required for the CO or hydrocarbon emissions from a PLASMOX<sup>®</sup> system treating NSCMP neutralents.

To identify any specific organic HAPs present in the air emissions from the PLASMOX<sup>®</sup> process, a GC-fingerprint analysis of the stack gas was conducted. The samples were collected using two different adsorbents in series. The first adsorbent was used for semi-volatile, the second for volatile compounds. In addition to the GC-fingerprint analysis, the stack gas was analyzed for acetaldehyde and formaldehyde (Table 6-8).

The results of the FID analyses of the stack gases from Test Runs GB-2 and HD-2 are included in Appendix A. The presence of HAPs in the stack gas may require monitoring of the stack gas. The tables in Appendix A show the semi-volatile and volatile organic compounds (SVOC and VOC) that were identified with sufficient probability in the stack gas. A compound was considered as identified if the correspondence between the mass-spectrum of the GC-MS-library and the measured spectrum was at least 80 %. 38

The FID data in Appendix A can be used in future tests of plasma destruction technologies as a guide as to which compounds are likely to be found in the stack gas. Sampling and analysis programs can then be developed to quantify these compounds and assess whether they are likely to exceed the EPA HAP major source thresholds, or state/local air toxic regulations.

#### 7.0 CONCLUSIONS

Test data and observations from the six test runs completed as part of the Limited Engineering Scale Testing of the PLASMOX® Technology were evaluated in accordance with the test criteria. Test conclusions based on these criteria are summarized below.

- The test data show that a PLASMOX<sup>®</sup> unit equivalent in size to the RIF-2 unit could process more than 14,000 gallons of NSCMP neutralent per year, assuming the demonstrated 13 L/hr processing rate and 50% availability. The maximum continuous flow demonstrated on MEA-based neutralent simulant was equivalent to 82 gallons/day.
- The RIF-2 unit operated continuously for all but one of the test runs. The unit was stopped prematurely due to a liquid feed system disturbance during a work-up run prior to the commencement of test run data collection. Aspirating air introduced with the liquid feed upset the control of the torch electrode. During test run operations, Test Run GB-2 was aborted after 6 hours of operation because of slag deposits on the crucible rim that caused it to bind against the reactor lid. The test was scheduled for 12 hours. Both incidents were caused by non-standard operating conditions. The liquid feed system was modified to eliminate the use of aspirated air. The location of slag feed was changed after Test Run GB-2. Both of these operational changes eliminated further upsets. For the remainder of the test program the system operated without incident and without significant operator interaction. The predominant operator activity consisted of regular recording of process conditions. However, the crucible binding incident highlights the need for design review of rotating crucible plasma systems.
- Improvements in the gas scrubber system are required. Test data indicate that the RIF-2 scrubbers are ineffective in removing acid gases, particulates and other compounds. Improved scrubbers will reduce the load on the HEPA filters, helping the system to meet regulatory emission limits.
- The data available to date indicate that the effluent streams from a PLASMOX<sup>®</sup> unit treating NSCMP neutralent will likely be disposed as non-hazardous wastes. Confirmation of these results are in progress. A report addendum will be issued when this data is available.
- Insufficient analytical data is currently available to complete a phosphorus balance. Additional analyses have been initiated in order to estimate the disposition of phosphorus throughout the RIF-2 system. A report addendum will be issued when this data is available. Preliminary analysis of the solid material collected from the reactor exit piping indicate that the phosphorus may be plating out inside the system. A preliminary analysis of stack gas sampling condensate from Test Run GB-1 also indicate that phosphorus may be leaving the system via the stack gas. Chemical analysis of material deposited in the reactor exit piping after Test Run HD-1 indicated the presence of silicon, phosphorus and chlorine. The mechanism for the formation of this deposited material is unclear. The presence of silicon and phosphorus in the deposited solid material demonstrates that silicon is depleted from the slag and deposits in the system and that phosphorus also deposits in the system. The high concentration of particulate material in the stack gas is

likely a result of slag volatilization and condensation – no data has been supplied that would support the assumption that particulate loading results from refractory conditioning. It is possible that entrainment and deposition of the slag material is attributable to specific system design features, such as insufficient disengagement space in the reactor. Testing of other plasma systems should investigate the design issues that may contribute to this effect.

- Test data have shown that NO<sub>x</sub> emissions, carbon monoxide, hydrocarbons and organic Hazardous Air Pollutants are all below regulatory limits. Testing of other plasma systems should focus on the ability of properly designed off-gas treatment systems to reduce particulate and metal concentrations to below MACT limits. Similarly, future tests of plasma systems that utilize a ROC or secondary combustor should focus on combustor process control to reduce potential formation of dioxins/furans. The PLASMOX<sup>®</sup> system tests could not confirm whether detectable levels of dioxins/furans were a result of equipment contamination.
- The MGC electrode and torch designs provide significant improvements over competing water-cooled electrode torch designs in terms of reliability and safety. Although the PLASMOX® electrode and torch reduce the risk of releasing torch cooling water that could cause pressure excursions, it would be prudent to include expansion tanks for any NSCMP applications. In addition, significant improvements to the efficacy of the unit can be made by optimizing the feed locations, designing the ROC for the specific feeds being processed and by sizing the Off-Gas Treatment System for processing of liquid feeds.

#### 8.0 RECOMMENDATIONS

- It is recommended that a plasma system be tested at the pilot scale on NSCMP liquid and solid waste streams. These tests should be prototypical of full-scale operation.
- Pilot scale testing of a plasma system should investigate materials of construction impacts
  of the various NSCMP waste streams and the long-term effect of, and mechanism of
  deposition of material in the system.
- Additional data to support permitting of a plasma system should be collected during pilot scale testing.
- A survey of plasma system technology providers indicates that there are more than a
  dozen potential competitive sources of plasma systems. Additional testing of plasma
  technology should capitalize on the availability of competing designs to address the
  technical issues identified in these tests.

#### 9.0 REFERENCES

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<sup>&</sup>lt;sup>3</sup> Technology Evaluation Panel/Stone & Webster Engineering Corporation, <u>Evaluation of Neutralent Post-Treatment Technologies for the Non-Stockpile Chemical Materiel Program, Contract No. DAAM-01-96-D-0010, September 19, 2000.</u>

<sup>&</sup>lt;sup>4</sup> National Research Council, <u>Evaluation of Demonstration Test Results of Alternative Technologies for Demilitarization of Assembled Chemical Weapons – A Supplemental Review</u>, National Academy Press, Washington, DC, 2000.

<sup>&</sup>lt;sup>5</sup> Stone & Webster Engineering Corporation, <u>Evaluation of Technologies Proposed Under the Broad Agency Announcement BAA-97-ACWA-1</u>, Contract No. DAAM-01-96-D-0010, March 2000. PROCUREMENT SENSITIVE MATERIAL

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<sup>&</sup>lt;sup>8</sup> Utah Division of Solid and Hazardous Waste, <u>MMD-1</u>, <u>Research Development and</u> Demonstration RCRA Permit, Issued May 14, 1999, Appendix 4A.

<sup>&</sup>lt;sup>9</sup> Utah Division of Solid and Hazardous Waste, Appendix 4B, p. 12.

<sup>&</sup>lt;sup>10</sup> Dr. Graf AG, <u>Emission Measurements During Demonstration Test of the PLASMOX System</u>, Report No. 92.3949, March 2001.

<sup>&</sup>lt;sup>11</sup> Dr. Graf AG, <u>Emission Measurements During Demonstration Test of the PLASMOX System</u>, Report No. 92.3949, March 2001.

<sup>&</sup>lt;sup>12</sup> Burns and Roe, <u>Limited Engineering Scale Testing of the Plasmox Technology to Treat chemical Warfare Material – Final Report, March 2001.</u>

<sup>&</sup>lt;sup>13</sup> Dr. Graf AG, <u>Emission Measurements During Demonstration Test of the PLASMOX System</u>, Report No. 92.3949, March 2001.

<sup>&</sup>lt;sup>14</sup> Dr. Graf AG, Emission Measurements During Demonstration Test of the PLASMOX System, Report No. 92.3949, March 2001.

<sup>&</sup>lt;sup>15</sup> Dr. Graf AG, Emission Measurements During Demonstration Test of the PLASMOX System, Report No. 92.3949, March 2001.

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- <sup>20</sup> Burns and Roe Enterprises, Inc., <u>TEST PLAN Limited Engineering Scale Testing of the PLASMOX Technology to Treat Chemical Warfare Material</u>, 4 January 2001.
- <sup>21</sup> Burns and Roe, <u>Limited Engineering Scale Testing of the Plasmox Technology to Treat</u> Chemical Warfare Material Final Report, March 2001, Figure 5.2.5-1.
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- <sup>24</sup> Burns and Roe, <u>Limited Engineering Scale Testing of the Plasmox Technology to Treat Chemical Warfare Material Final Report</u>, March 2001, Section 5.2.3.2.
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- <sup>26</sup> NPDES Permit No. AR0001678, Part I, Section A: Outfall 002
- <sup>27</sup> Burns and Roe, <u>Limited Engineering Scale Testing of the Plasmox Technology to Treat Chemical Warfare Material Final Report</u>, March 2001, Section 5.2.3.5.
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- <sup>29</sup> Dr. Graf AG, <u>Emission Measurements During Demonstration Test of the PLASMOX System</u>, Report No. 92.3949, March 2001.
- <sup>30</sup> Code of Federal Regulations, Title 40, Part 60, Subpart CCCC.
- <sup>31</sup> Burns and Roe, <u>Limited Engineering Scale Testing of the Plasmox Technology to Treat Chemical Warfare Material Final Report</u>, March 2001, Section 5.2.3.1.
- $^{32}~ppm_{v,d} = (mg/m^3)~x~(1~x~10^3)~x~(0.0224~m^3/mole)~/~MW~(g/mole)~x~(20.9-7\%~O_2)~/~(20.9-corrected~\%O_2)$
- <sup>33</sup> Dr. Graf AG, <u>Emission Measurements During Demonstration Test of the PLASMOX System</u>, Report No. 92.3949, March 2001.
- <sup>34</sup> Code of Federal Regulations, Title 40, Part 63, Subpart EEE.

<sup>&</sup>lt;sup>17</sup> Burns and Roe Enterprises, Inc., <u>TEST PLAN – Limited Engineering Scale Testing of the PLASMOX Technology to Treat Chemical Warfare Material</u>, 4 January 2001.

<sup>&</sup>lt;sup>35</sup> Burns and Roe, <u>Limited Engineering Scale Testing of the Plasmox Technology to Treat Chemical Warfare Material – Final Report, March 2001, Section 5.2.3.1.</u>

<sup>&</sup>lt;sup>36</sup> Code of Federal Regulations, Title 40, Part 63, Subpart EEE.

<sup>&</sup>lt;sup>37</sup> Code of Federal Regulations, Title 40, Part 63, Subpart EEE.

<sup>&</sup>lt;sup>38</sup> Dr. Graf AG, <u>Emission Measurements During Demonstration Test of the PLASMOX</u> System, Report No. 92.3949, March 2001.

## APPENDIX A

## **Analytical Results**

## MGC-Plasma AG, Muttenz

# **Emission Measurements During Demonstration Tests**of the PLASMOX System

Report no. : 92.3949

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## 1. Introduction

As ordered by MGC-Plasma Ltd, Muttenz, Switzerland, the following measurements have been performed at the PLASMOX system in Muttenz during six days of demonstration tests of simulated chemical weapon chemicals:

## Test parameters for stack testing (behind ventilator)

### Continuously measured parameters (during all tests):

- Oxygen by paramagnetic detector
- Carbon dioxide by infrared detector
- Carbon monoxide by infrared detector
- Nitrogen oxides by chemiluminescence detector
- Hydrocarbons by flame ionisation detector

Analyses of the system showed that the stack gas has to be diluted with heated ambient air to prevent condensation in the gas duct. Therefore heated, cleaned air was added before the guard filter box (HEPA filter). To be able to correct for this dilution air, an additional oxygen measurement was installed behind the scrubber and before the dilution air inlet.

## Grab sampling during test GB-1 (12.1.2001)

- Particulates (one sample, 3 h sampling time)
- Metals: copper, chromium, iron and zinc; particle bound and volatile parts, one sample, 3 h sampling time
- Hydrocyanic acid (one sample, 3 h sampling time)
- Aldehydes (one sample, 1 h sampling time)
- Monoethanolamine (one sample, 2 h sampling time)
- Dimethyl-methylphosphonate (one sample, 3 h sampling time)
- Identification of SVOC's and VOC's (by GC-MS-Fingerprint)

#### Grab sampling during test HD-2 (17.1.2001)

- Particulates (one sample, 3 h sampling time)
- Metals (copper, chromium, iron and zinc; particle bound and volatile parts, one sample, 3 h sampling time)
- Hydrocyanic acid (one sample, 3 h sampling time)
- Hydrochloric acid (one sample, 3 h sampling time)
- Aldehydes (one sample, 1 h sampling time)
- Monoethanolamine (one sample, 2 h sampling time)
- Dioxins and furans (one sample, 4 6 h sampling time)
- Identification of SVOC's and VOC's (by GC-MS-Fingerprint)

## Test parameters for grab sampling at additional testing locations Between PLASMOX system and rapid oxidation chamber (test GB-2, 13.1.2001)

- Monoethanolamine (one sample, 1 h sampling time)

## Between PLASMOX system and rapid oxidation chamber (test HD-2, 17.1.2001)

- Monoethanolamine (one sample, 1 h sampling time)

## Between rapid oxidation chamber and quenche (test HD-2, 17.1.2001)

- Dioxins and furans (one sample, 4 - 6 h sampling time)

## 2. Customer

MGC-Plasma AG Mr. H. Felix Hofackerstrasse 24 4132 Muttenz

## 3. Plant

A detailed description of the system is available from MGC-Plasma Ltd.

The system consists of the following parts:

- PLASMOX system heated by an arc-discharge between two electrodes and a crucible.
- Rapid oxidation chamber heated by an air-gas-burner
- Quenche to shock-cool the gas between the post-combustion chamber and the scrubber.
- Two-stage wet scrubber
- Inlet of electrically heated dilution air; for the tests an activated carbon filter was installed to eliminate organic contamination from the ambient air
- Filterbox for a guard filter (empty during these tests)
- Exhaust-ventilator
- Exhaust line to the chimney

## 4. Accomplishment of Measurements

The measurements were performed by Dr. M. Andrée (M.A.) and Mr. H.U. Bieri (H.U.B.) according to the following schedule (detailed schedule see MGC-Plasma Ltd.):

12.1.2001: Test GB-1, 12:00 to 13.1.2001 1:30 by M.A. and H.U.B.

13.1.2001: Test GB-2, 14:45 to 14.1.2001 1:30 by M.A.

15.1.2001: Test GB-3, 14:00 to 20:30 by H.U.B.

16.1.2001: Test HD-1, 15:30 to 22:30 by H.U.B.

17.1.2001: Test HD-2, 13:00 to 21:00 by M.A. and H.U.B.

18.1.2001: Test HD-3, 14:00 to 19.1.2001 2:00 by H.U.B.

## Measuring point between PLASMOX system and rapid oxidation chamber

Grab sampling of Monoethynoleamine:

1"-connection piece in straight metal tube PLASMOX system and rapid oxidation chamber; due to the high temperature of the gas (> 1'000°C) a special extraction line consisting of 1 m of ceramic tube (15 mm) was mounted on front of the adsoption tube.

## Measuring point between rapid oxidation chamber and quenche

Grab sampling of Dioxins and Furans:

1 EMPA-connection piece in the T-shaped piece immediately in front of the quenche. This sampling point is not conform to the rules. The probe was oriented directly against the gas flow (without 90° bend between probe and sampling orifice). The gas flow is not at all laminar as there is a 180° bend in front (approximately 1.5 m) and a 90° bend behind (approximately 0.5 m) the sampling point.

## Measuring point behind scrubber and before dilution air inlet

Continuous sampling of oxygen:

3/4"-connection piece in straight metal tube behind srubber and before dilution air inlet.

## Measuring points at the exhaust line

Grab sampling of Monoethynoleamine and for GC-MS-fingerprints:

3/4"-connection piece in rising metal tube behind exhaust ventilator.

Continuous sampling:

3/4"-connection piece in rising metal tube behind exhaust ventilator.

Grab sampling of DMMP and dioxins and furans:

1 horizontally oriented EMPA-connection piece in slightly tilted PVC-pipe before exhaust.

Grab sampling of particulates, aldehydes, HCl, HCN, metals:

1 vertically oriented EMPA-connection piece in slightly tilted PVC-pipe before exhaust.

## Operating conditions

The operating conditions were registered by the operators of MGC-Plasma Ltd.

## 5. Results of test GB-1 (12./13.1.2001)

#### Note:

All concentrations refer to dry gas at normal conditions (273 K, 101.3 kPa) and are normalised to the oxygen content of the gas before addition of dilution-air.

## 5.1. Measurements of the volumetric flow rate

Plant: PLASMOX system, sampling point: exhaust line Cross section at sampling point: ø 250 mm, Area: 0.049 m²

Time	Т	b	Δр	f*	٧	$V_b$	$V_{n,f}$	$V_{n,tr}$
	°C	mbar	mbar	kg/m³	m/s	m³/h	Nm³/h	Nm³/h
17:45	65	990	0.1	0.17	3.7	660	520	430 ± 60
21:20	53	990	0	0.11	2.8	490	400	$350 \pm 90$
Average						570	460	390 ± 70

<sup>\*</sup> average humidity during aldehyde respectively particulates measurements

## Remark:

The volumetric flow rate is kept stable by the system. A change in the flow rate at the exhaust is only possible, if the amount of dilution air is manually changed.

## 5.2. Measurements of O<sub>2</sub>, CO<sub>2</sub>, CO, NO<sub>2</sub> and HC

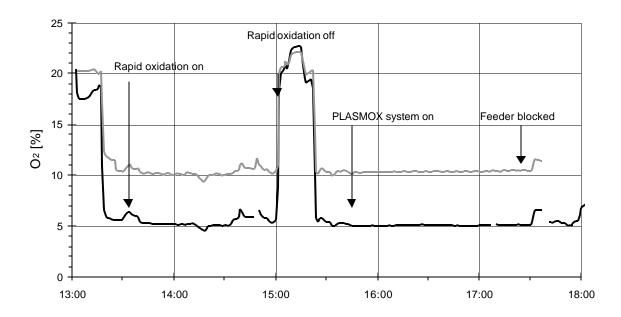
## a) Comparison of oxygen-content before and after dilution-air inlet

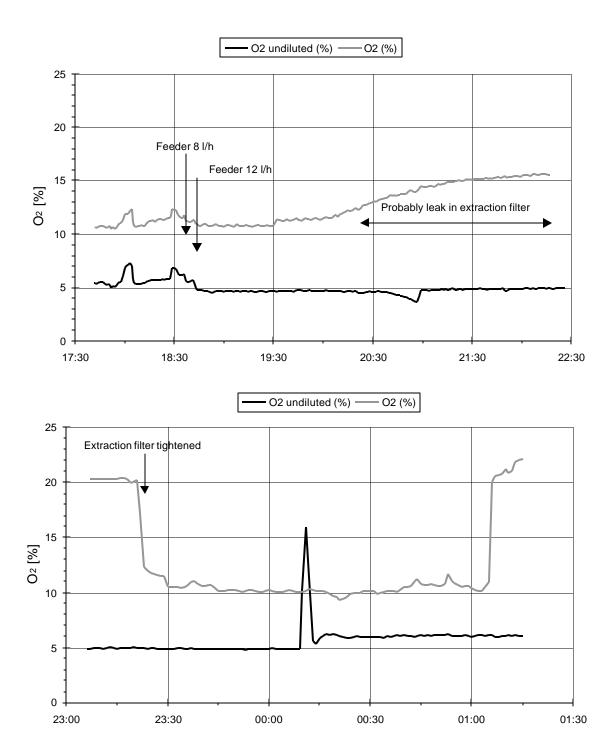
12.0	1.01	Before HEPA Behind HE	
Inte	erval	O <sub>2</sub> undiluted	O <sub>2</sub> diluted
from	til	%	%
13:30	14:30	$5.3 \pm 0.3$	10.1 ± 0.5
14:30	15:30	11.4 ± 0.6	14.3 ± 0.7
15:30	16:30	$5.1 \pm 0.3$	10.3 ± 0.5
16:30	17:30	$5.2 \pm 0.3$	10.4 ± 0.5
17:40	18:40	$5.8 \pm 0.3$	11.2 ± 0.6
18:40	19:40	$4.7 \pm 0.2$	10.9 ± 0.5
19:40	20:40	$4.6 \pm 0.2$	12.1 ± 0.6*
20:40	21:40	$4.6 \pm 0.2$	14.6 ± 0.7*
21:40	22:26	$4.8 \pm 0.2$	15.5 ± 0.8*
23:05	00:05	$4.9 \pm 0.2$	11.2 ± 0.6
00:05	01:05	$6.3 \pm 0.3$	10.6 ± 0.5
13:30	00:05	$5.7 \pm 0.3$	11.9 ± 0.6

<sup>\*</sup> values probably influeced by a tiny leak developping in the extraction filter of the continuous measurement. Therefore CO, NO<sub>2</sub> and HC had to be corrected for this effect. The other measured parameters were not influenced.

Grafic representation of the oxygen-content before and after dilution (time-base one minute)







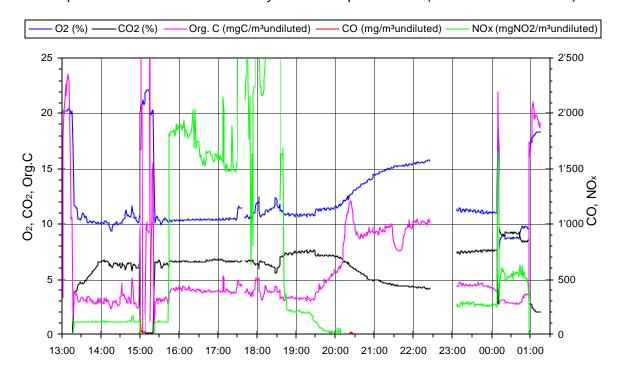
## b) Continuously measured concentrations in the exhaust line

Inte	rval	O <sub>2</sub>	CO <sub>2</sub>	CO *	NOx *	Org. C *
from	til	%	%	mg/m³	mgNO <sub>2</sub> /m³	mgC/m³
13:30	14:30	10.1 ± 0.5	$6.0 \pm 0.3$	<9	113 ± 15	3 ± 1
14:30	15:30	14.3 ± 0.7	$4.1 \pm 0.2$	<9	73 ± 15	6 ± 1
15:30	16:30	10.3 ± 0.5	$6.5 \pm 0.3$	<9	1'437 ± 160	4 ± 1
16:30	17:30	10.4 ± 0.5	$6.7 \pm 0.3$	<9	1'563 ± 174	4 ± 1
17:40	18:40	11.2 ± 0.6	$6.4 \pm 0.3$	<10	2'613 ± 301	4 ± 1
18:40	19:40	10.9 ± 0.5	$7.4 \pm 0.4$	<9	$198 \pm 23$	3 ± 1
19:40	20:40	12.1 ± 0.6	$6.6 \pm 0.3$	<11	<18	7 ± 1

Inte	rval	O <sub>2</sub> CO <sub>2</sub> CO*		CO *	NOx *	Org. C *
from	til	%	%	mg/m³	mgNO <sub>2</sub> /m³	mgC/m³
20:40	21:40	14.6 ± 0.7	4.8 ± 0.2	<15	<24	8 ± 2
21:40	22:26	15.5 ± 0.8	$4.3 \pm 0.2$	<17	<28	9 ± 2
23:05	00:05	11.2 ± 0.6	$7.5 \pm 0.4$	<10	$269 \pm 31$	4 ± 1
00:05	01:05	10.6 ± 0.5	$7.8 \pm 0.4$	<10	$493 \pm 55$	4 ± 1
13:30	01:05	11.9 ± 0.6	$6.2 \pm 0.3$	<11	634 ± 76	5 ± 1

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

Grafic representation of the continuously measured parameters (time-base one minute)



## 5.3. Measurements of particulates and metals

#### Measured concentrations

Samplin	Sampling interval Particulates		Chromium	Copper	Iron	Zinc
from	til	mg/m³	mg/m³	mg/m³	mg/m³	mg/m³
22:00	01:00	170 ± 20	$0.99 \pm 0.20$	$0.094 \pm 0.019$	$0.090 \pm 0.017$	0.094 ± 0.018

the concentrations are corrected for the dilution-air effect

#### Calculated mass flows

Sampling interval		Particulates	Chromium	Copper	Iron	Zinc
from	til	g/h	g/h	g/h	g/h	g/h
22:00	01:00	43 ± 9	$0.25 \pm 0.07$	0.024 ± 0.007	$0.023 \pm 0.006$	0.024 ± 0.006

The partition of the metals into a dust-bound and a filter-passing part is shown in the detailed results in appendix 1.

## 5.4. Measurements of aldehydes, HCN and monoethanoleamine

#### Measured concentrations

Sampling interval		Acetaldehyde	Formaldehyde	Sum	$O_2$
from til		mg/m³	mg/m³	mg/m³	%
22:00	01:00	0.41 ± 0.08	$0.33 \pm 0.07$	$0.74 \pm 0.11$	10.9 ± 0.5

Sampling	mpling interval HCN		MEA	O <sub>2 (MEA)</sub>
from	til	mg/m³	mg/m³	%
22:00	01:00	< 1.6	< 0.2	11.1 ± 0.6

the concentrations are corrected for the dilution-air effect

#### Calculated mass flows

Sampling	Sampling interval		Acetaldehyde Formaldehyde	
from	til	g/h	g/h	g/h
22:00	01:00	0.10 ± 0.03	0.08 ± 0.02	0.19 ± 0.04

Samplin	g interval	HCN	MEA
from	til	g/h	g/h
22:00	01:00	< 0.4	< 0.04

#### Remark:

Detailed results are given in appendix 2.

## 5.5. Measurements of dimethyl-methylphosphonate

Measured concentration

Sampling interval		V	m	C <sub>n</sub> *
from	til	m³	mg	mg/m³
22:05	01:05	0.372	< 0.17	< 0.7

#### Calculated mass flow

DMMP	
g/h	
< 0.2	

## 5.6. Identification of organic compounds (VOC's and SVOC's)

The samples are taken using two different adsorbents in series. The first adsorbent is suited for semi-volatile, the second for volatile compounds. The two parts of the sample are analysed separately.

\* the concentration is corrected for the dilution-air effect

The following tables show the compounds which are identified with sufficient probability. A compound is considered as identified, if the correspondence between the mass-spectrum of the GC-MS-library and the measured spectrum is at least 80 %.

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## Adsorbent for semi-volatile compounds

Identified compound	CAS-number	Percentage of total peak area
Phenanthrene	85-01-8	19.1%
Xylenes		7.8%
Hexane	110-54-3	6.5%
Toluene	108-88-3	4.8%
Diisobutylphthalate	84-69-5	4.6%
Naphthalene	91-20-3	4.0%
Fluoranthene	206-44-0	3.7%
9H-fluorene-9-one	486-25-9	3.0%
Benzene	71-43-2	2.9%
Acetic acid ethyl ester	141-78-6	2.7%
Dibenzothiophene	132-65-0	2.5%
Etyhlbenzene	100-41-4	2.5%
1,1-biphenyl	92-52-4	2.3%
4-methyl-dibenzofuran	7320-53-8	2.1%
Hexachlorobenzene	118-74-1	2.0%
Dibenzofuran	132-64-9	2.0%
Ethanole	64-17-5	2.0%
Methylnaphthalenes		1.7%
1,X-dichloro-benzene		1.7%
2-propanole	67-63-0	1.4%
4H-cyclopenta[def]phenanthrene	203-64-5	1.3%
2-phenylnaphthalene	35465-71-5	1.2%
Pentachlorobenzene	608-93-5	1.1%
X-methyl-1,1-biphenyl		1.0%
Methyl-biphenyl		0.9%
Chloroform	67-66-3	0.8%
Pyrene	129-00-0	0.7%
2,6-xx-2,5-cyclohexadiene-1,4-dione	719-22-2	0.7%
Octamethyl-cyclotetrasiloxane	556-67-2	0.6%
9H-fluorene	86-73-7	0.6%
Benzaldehyde		0.6%
1-ethyl-naphthalene	1127-76-0	0.3%
Sum		88.8%

## Adsorbent for volatile compounds

Identified compound	CAS-number	Percentage of total peak area
Diisobutylphthalate	84-69-5	8.6%
Ethyl-benzene	100-41-4	0.7%
Acetic acid	64-19-7	0.6%
Phenanthrene	85-01-8	0.5%
Xylenes		0.3%
1,X-dichloro-benzene		0.3%
Fluoranthene	206-44-0	0.2%
Sum		11.2%

#### Remarks:

The given percentage of the total peak area indicates the part of the compound relative to the total area of the chromatogram. This is a qualitative information regarding the main compounds of the mixture. It isn't possible to transform this information into a quantitative measure, as the analysator is not calibrated for the individual compounds. Therefore the two different adsorbents of the same sample cannot be compared, as the total area of the two samples maybe completely different.

## 6. Results of test GB-2 (13.1.2001)

#### Note:

All concentrations refer to dry gas at normal conditions (273 K, 101.3 kPa) and are normalised to the oxygencontent of the gas before addition of dilution-air.

## 6.1. Measurement of monoethanoleamine before rapid oxidation

Measured concentration

Sampling interval		V	m	C <sub>n</sub> *
from	til	m³	mg	mg/m³
15:28	16:38	0.018	< 0.002	< 0.1

Calculated mass flow

MEA
g/h
not available

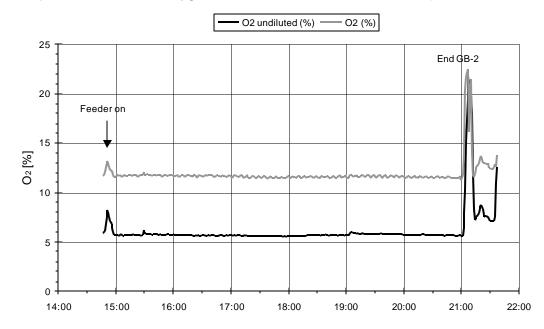
the concentration is given for the oxygen-content at the sampling point (not determined)

## 6.2. Measurements of O<sub>2</sub>, CO<sub>2</sub>, CO, NO<sub>2</sub> and HC in the exhaust line

## a) Comparison of oxygen-content before and after dilution-air inlet

13.01.01		Before HEPA	Behind HEPA
Inte	erval	O <sub>2</sub> undiluted	O <sub>2</sub> diluted
from	til	%	%
14:45	15:45	$5.9 \pm 0.3$	11.8 ± 0.6
15:45	16:45	$5.7 \pm 0.3$	11.7 ± 0.6
16:45	17:45	$5.6 \pm 0.3$	11.6 ± 0.6
17:45	18:45	$5.6 \pm 0.3$	11.6 ± 0.6
18:45	19:45	$5.8 \pm 0.3$	11.6 ± 0.6
19:45	20:45	$5.7 \pm 0.3$	11.6 ± 0.6
20:45	21:45	$8.8 \pm 0.4$	$13.7 \pm 0.7$
14:45	21:45	6.1 ± 0.3	11.9 ± 0.6

## Grafic representation of the oxygen-content before and after dilution (time-base one minute)

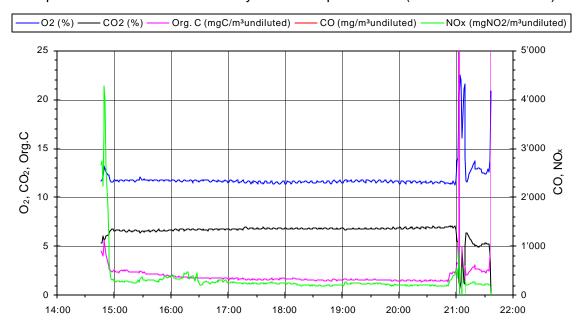


## b) Continuously measured concentrations in the exhaust line

Inte	erval	O <sub>2</sub>	CO <sub>2</sub>	CO*	NOx *	Org. C *
from	til	%	%	mg/m³	$mgNO_2/m^3$	mgC/m³
14:45	15:45	11.8 ± 0.6	$6.4 \pm 0.3$	<10	627 ± 75	3 ± 1
15:45	16:45	11.7 ± 0.6	$6.6 \pm 0.3$	<10	$331 \pm 39$	2 ± 1
16:45	17:45	11.6 ± 0.6	$6.8 \pm 0.3$	<10	$256 \pm 34$	2 ± 1
17:45	18:45	11.6 ± 0.6	$6.8 \pm 0.3$	<10	$218 \pm 33$	2 ± 1
18:45	19:45	11.6 ± 0.6	$6.8 \pm 0.3$	<10	$227 \pm 33$	2 ± 1
19:45	20:45	11.6 ± 0.6	$6.9 \pm 0.3$	<9	$213 \pm 31$	2 ± 1
20:45	21:45	13.7 ± 0.7	$5.2 \pm 0.3$	<12	191 ± 38	2 ± 1
14:45	21:45	11.9 ± 0.6	$6.5 \pm 0.3$	<10	297 ± 35	2 ± 1

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

## Grafic representation of the continuously measured parameters (time-base one minute)



## 7. Results of test GB-3 (15.1.2001)

## Note:

All concentrations refer to dry gas at normal conditions (273 K, 101.3 kPa) and are normalised to the oxygencontent of the gas before addition of dilution-air.

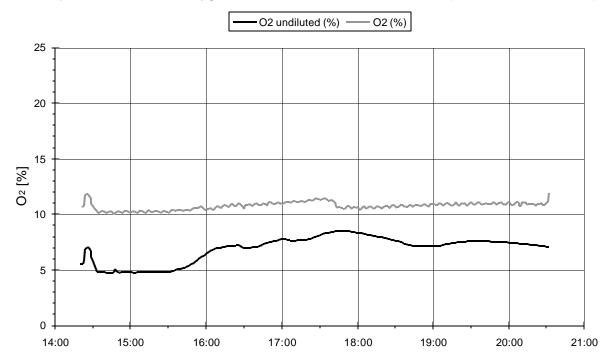
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## 7.1. Measurements of O<sub>2</sub>, CO<sub>2</sub>, CO, NO<sub>2</sub> and HC

## a) Comparison of oxygen-content before and after dilution-air inlet

15.01.01		Before HEPA	Behind HEPA
Inte	rval	O <sub>2</sub> undiluted	O <sub>2</sub> diluted
from	til	%	%
14:20	15:20	$5.0 \pm 0.3$	10.4 ± 0.5
15:20	16:20	$5.9 \pm 0.3$	10.5 ± 0.5
16:20	17:20	$7.4 \pm 0.4$	11.0 ± 0.5
17:20	18:20	$8.2 \pm 0.4$	10.9 ± 0.5
18:20	19:20	$7.4 \pm 0.4$	10.8 ± 0.5
19:20	20:20	$7.5 \pm 0.4$	10.9 ± 0.5
14:20	20:20	$6.9 \pm 0.3$	10.7 ± 0.5

Grafic representation of the oxygen-content before and after dilution (time-base one minute)

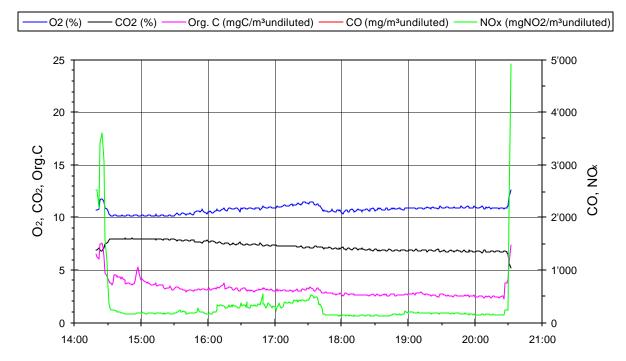


## b) Continuously measured concentrations in the exhaust line

Inte	rval	O <sub>2</sub>	CO <sub>2</sub>	CO*	NOx *	Org. C *
from	til	%	%	mg/m³	$mgNO_2/m^3$	mgC/m³
14:20	15:20	10.4 ± 0.5	$7.8 \pm 0.4$	<9	530 ± 59	4 ± 1
15:20	16:20	10.5 ± 0.5	$7.7 \pm 0.4$	<9	$216 \pm 30$	3 ± 1
16:20	17:20	11.0 ± 0.5	$7.4 \pm 0.4$	<10	$348 \pm 40$	3 ± 1
17:20	18:20	10.9 ± 0.5	$7.1 \pm 0.4$	<9	246 ± 31	3 ± 1
18:20	19:20	10.8 ± 0.5	$6.9 \pm 0.3$	<9	$166 \pm 30$	3 ± 1
19:20	20:20	10.9 ± 0.5	$6.8 \pm 0.3$	<9	177 ± 29	3 ± 1
14:20	20:20	10.7 ± 0.5	$7.3 \pm 0.4$	<9	280 ± 32	3 ± 1

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

Grafic representation of the continuously measured parameters (time-base one minute)



## 8. Results of test HD-1 (16.1.2001)

#### Note:

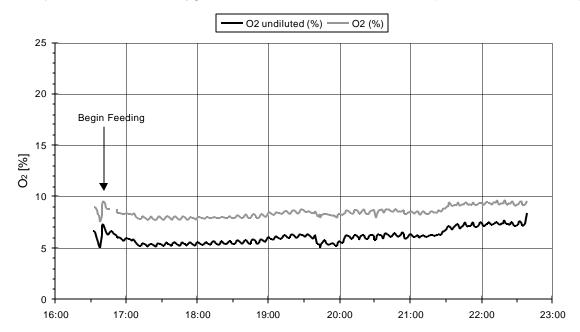
All concentrations refer to dry gas at normal conditions (273 K, 101.3 kPa) and are normalised to the oxygencontent of the gas before addition of dilution-air.

## 8.1. Measurements of O<sub>2</sub>, CO<sub>2</sub>, CO, NO<sub>2</sub> and HC

## a) Comparison of oxygen-content before and after dilution-air inlet

	16.01.01		Before HEPA	Behind HEPA
	Inte	erval	O <sub>2</sub> undiluted	O <sub>2</sub> diluted
	from	til	%	%
	16:31	17:00	$6.2 \pm 0.3$	10.5 ± 0.5
	17:00	18:00	$5.4 \pm 0.3$	$8.0 \pm 0.4$
	18:00	19:00	$5.6 \pm 0.3$	$8.0 \pm 0.4$
	19:00	20:00	$5.9 \pm 0.3$	8.4 ± 0.4
ĺ	20:00	21:00	$6.1 \pm 0.3$	8.5 ± 0.4
	21:00	22:00	$6.7 \pm 0.3$	$8.9 \pm 0.4$
	22:00	22:30	$7.4 \pm 0.4$	9.4 ± 0.5
	16:31	22:30	6.1 ± 0.3	$8.6 \pm 0.4$

Grafic representation of the oxygen-content before and after dilution (time-base one minute)

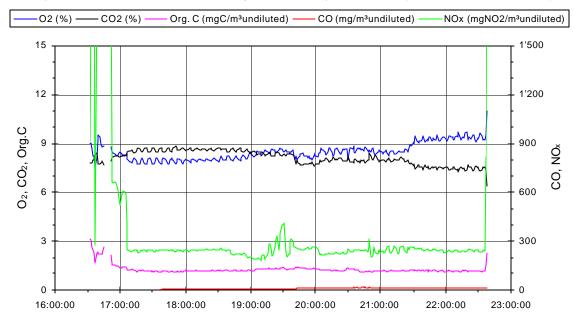


## b) Continuously measured concentrations in the exhaust line

Inte	rval	O <sub>2</sub>	CO <sub>2</sub>	CO *	NOx *	Org. C *
from	til	%	%	mg/m³	mgNO <sub>2</sub> /m³	mgC/m³
16:31	17:00	10.5 ± 0.5	$6.8 \pm 0.3$	<9	1'499 ± 167	3 ± 1
17:00	18:00	$8.0 \pm 0.4$	$8.6 \pm 0.4$	<7	$273 \pm 29$	1 ± 1
18:00	19:00	$8.0 \pm 0.4$	$8.6 \pm 0.4$	<7	$236 \pm 25$	1 ± 1
19:00	20:00	$8.4 \pm 0.4$	$8.2 \pm 0.4$	<8	$253 \pm 27$	1 ± 1
20:00	21:00	$8.5 \pm 0.4$	$8.0 \pm 0.4$	$16 \pm 7$	$233 \pm 25$	1 ± 1
21:00	22:00	$8.9 \pm 0.4$	$7.7 \pm 0.4$	$16 \pm 7$	$248 \pm 26$	1 ± 1
22:00	22:30	$9.4 \pm 0.5$	$7.5 \pm 0.4$	$13 \pm 7$	$239 \pm 26$	1 ± 1
16:31	22:30	$8.6 \pm 0.4$	$8.0 \pm 0.4$	11 ± 8	$349 \pm 37$	1 ± 1

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

Grafic representation of the continuously measured parameters (time-base one minute)



## 9. Results of test HD-2 (17.1.2001)

#### Note:

All concentrations refer to dry gas at normal conditions (273 K, 101.3 kPa) and are normalised to the oxygencontent of the gas before addition of dilution-air.

## 9.1. Measurements of the volumetric flow rate

Plant: PLASMOX system, sampling point: exhaust line Cross section at sampling point: ø 250 mm, Area: 0.049 m²

Time	Т	b	Δρ	f*	٧	$V_b$	$V_{n,f}$	$V_{n,tr}$
	°C	mbar	mbar	kg/m³	m/s	m³/h	Nm³/h	Nm³/h
14:05	57	985	0.1	0.11	3.5	610	490	440 ± 70

<sup>\*</sup> average humidity during the dioxine- and furane-measurements

## Remark:

The volumetric flow rate is kept stable by the system. A change in the flow rate at the exhaust is only possible, if the amount of dilution air is manually changed.

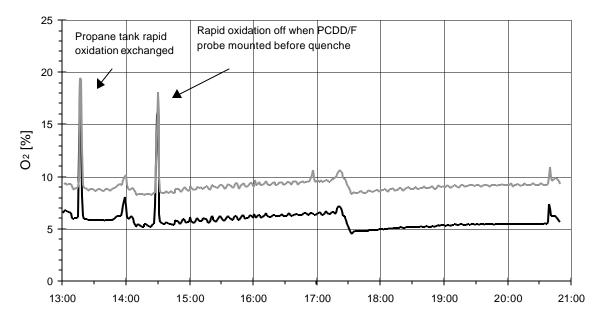
## 9.2. Measurements of O<sub>2</sub>, CO<sub>2</sub>, CO, NO<sub>2</sub> and HC

## a) Comparison of oxygen-content before and after dilution-air inlet

17.01	.2001	Before HEPA	Behind HEPA
Inte	rval	O <sub>2</sub> undiluted	O <sub>2</sub> diluted
from	til	%	%
13:00	14:00	$6.6 \pm 0.3$	$9.4 \pm 0.5$
14:00	15:00	$6.0 \pm 0.3$	$8.9 \pm 0.4$
15:00	16:00	$6.0 \pm 0.3$	9.1 ± 0.5
16:00	17:00	$6.3 \pm 0.3$	$9.4 \pm 0.5$
17:00	18:00	$5.6 \pm 0.3$	$9.1 \pm 0.5$
18:00	19:00	$5.2 \pm 0.3$	$8.9 \pm 0.4$
19:00	20:00	$5.4 \pm 0.3$	$9.1 \pm 0.5$
20:00	20:49	$5.7 \pm 0.3$	$9.4 \pm 0.5$
13:00	20:49	5.9 ± 0.3	$9.2 \pm 0.5$

Grafic representation of the oxygen-content before and after dilution (time-base one minute)



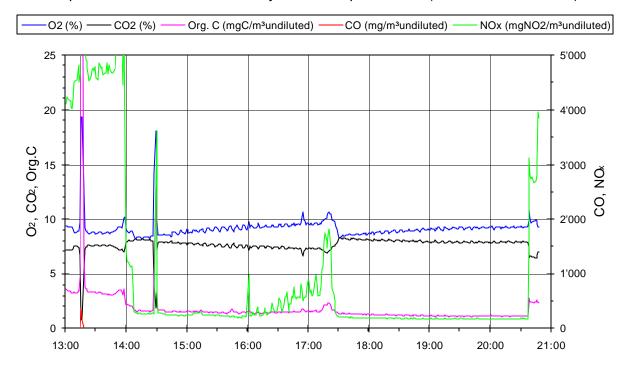


## b) Continuously measured concentrations in the exhaust line

Inte	rval	O <sub>2</sub>	CO <sub>2</sub>	CO *	NOx *	Org. C *
from	til	%	%	mg/m³	mgNO <sub>2</sub> /m³	mgC/m³
13:00	14:00	$9.4 \pm 0.5$	$7.2 \pm 0.4$	<9	4'926 ± 531	4 ± 1
14:00	15:00	$8.9 \pm 0.4$	$7.7 \pm 0.4$	<9	$404 \pm 43$	2 ± 1
15:00	16:00	$9.1 \pm 0.5$	$7.6 \pm 0.4$	<8	$233 \pm 28$	1 ± 1
16:00	17:00	$9.4 \pm 0.5$	$7.4 \pm 0.4$	<8	$437 \pm 47$	1 ± 1
17:00	18:00	$9.1 \pm 0.5$	$7.7 \pm 0.4$	<8	$532 \pm 57$	2 ± 1
18:00	19:00	$8.9 \pm 0.4$	$8.0 \pm 0.4$	<8	$188 \pm 26$	1 ± 1
19:00	20:00	$9.1 \pm 0.5$	$7.9 \pm 0.4$	<8	182 ± 26	1 ± 1
20:00	20:49	$9.4 \pm 0.5$	$7.6 \pm 0.4$	<8	$819 \pm 88$	1 ± 1
13:00	20:49	9.2 ± 0.5	$7.6 \pm 0.4$	<8	969 ± 104	2 ± 1

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

## Grafic representation of the continuously measured parameters (time-base one minute)



## 9.3. Measurements of particulates and metals

## Measured concentrations

Samplin	g interval	Particulates	Chromium	Copper	Iron	Zinc
from	til	mg/m³	mg/m³	mg/m³	mg/m³	mg/m³
16:45	19:30	270 ± 30	$0.36 \pm 0.07$	$0.62 \pm 0.13$	$0.14 \pm 0.03$	$2.9 \pm 0.6$

the concentrations are corrected for the dilution-air effect

#### Calculated mass flows

Samplin	g interval	Particulates	Chromium	Copper	Iron	Zinc
from	til	g/h	g/h	g/h	g/h	g/h
16:45	19:30	90 ± 17	0.12 ± 0.03	0.21 ± 0.05	0.05 ± 0.01	1.0 ± 0.3

The partition of the metals into a dust-bound and a filter-passing part is shown in the detailed results in appendix 3.

## 9.4. Measurement of monoethanoleamine

## a) Measurement between PLASMOX system and rapid oxidation chamber

Measured concentration

Sampling	Sampling interval		m	C <sub>n</sub> *
from	til	m³	mg	mg/m³
17:13	19:13	0.024	< 0.002	< 0.1

Calculated mass flow

MEA	I
g/h	
not available	

<sup>\*</sup> the concentration is expressed at the oxygen-content at the sampling point

## b) Measurement at the exhaust line

#### Measured concentration

Sampling	Sampling interval		m	C <sub>n</sub> *	
from	til	m³	mg	mg/m³	
17:10	19:10	0.022	< 0.004	< 0.2	

#### Calculated mass flow

MEA	O <sub>2</sub>		
g/h	%		
< 0.08	9.0 ± 0.5		

<sup>\*</sup> the concentration is corrected for the dilution-air effect

## 9.5. Measurements of aldehydes, HCI and HCN

## Measured concentrations

Sampling interval		Acetaldehyde	Formaldehyde	Sum	O <sub>2</sub>
from	til	mg/m³	mg/m³	mg/m³	%
15:30	16:30	0.17 ± 0.03	0.21 ± 0.04	$0.38 \pm 0.06$	9.2 ± 0.5

Sampling interval		HCN	HCI	O <sub>2</sub>
from	til	mg/m³	mg/m³	%
16:45	19:30	< 2	$0.36 \pm 0.04$	9.1 ± 0.5

the concentrations are corrected for the dilution-air effect

#### Calculated mass flows

Sampling interval		Acetaldehyde	Formaldehyde	Sum
from	til	g/h	g/h	g/h
15:30	16:30	0.06 ± 0.01	0.07 ± 0.02	0.13 ± 0.02

Samplin	g interval	HCN	MEA
from	til	g/h	g/h
16:45	19:30	< 0.6	0.12 ± 0.02

#### Remark:

Detailed results are given in appendix 4.

## 9.6. Identification of organic compounds (VOC's and SVOC's)

The samples are taken using two different adsorbents in series. The first adsorbent is suited for semi-volatile, the second for volatile compounds. The two parts of the sample are analysed separately.

The following tables show the compounds which are identified with sufficient probability. A compound is considered as identified, if the correspondence between the mass-spectrum of the GC-MS-library and the measured spectrum is at least  $80\,\%$ .

Identified compound	CAS- number	Percentage of total peak area			
Phenanthrene	85-01-8	26.6%			
Acetaldehyde	75-07-0	7.3%			
Fluoranthene	206-44-0	7.1%			
Xylenes		6.5%			
Naphthalene	91-20-3	4.2%			
4-methyl-dibenzofuran	7320-53-8	3.2%			
Dibenzothiophene	132-65-0	3.1%			
Dibenzofuran	132-64-9	2.9%			
Methylnaphthalenes		2.6%			
Hexachlorobenzene	118-74-1	2.5%			
1,1-biphenyl	92-52-4	2.2%			
9H-fluorene-9-one	486-25-9	2.0%			
Etyhlbenzene	100-41-4	1.8%			
4H-cyclopenta[def]phenanthrene	203-64-5	1.8%			
2,6-xx-2,5-cyclohexadiene-1,4-dione	719-22-2	1.7%			
1,3-dichloro-benzene	541-73-1	1.6%			
Pyrene	129-00-0	1.6%			
2-phenylnaphthalene	35465-71-5	1.5%			
Decane	124-18-5	1.4%			
Hexane	110-54-3	1.4%			
Pentachlorobenzene	608-93-5	1.3%			
1H-fluorene	86-73-7	1.2%			
1-phenylnaphthalene	605-02-7	1.1%			
Diisobutylphthalate	84-69-5	1.1%			
Octamethyl-cyclotetrasiloxane	556-67-2	1.0%			
4-methyl-1,1-biphenyl	644-08-6	0.9%			
1-ethyl-3-methyl-benzene	620-14-4	0.7%			
Undecane	1120-21-4	0.7%			
1-ethyl-naphthalene	1127-76-0	0.4%			
Sum	Sum				

Identified compound	CAS- number	Percentage of total peak area
Diisobutylphthalate	84-69-5	2.1%
Phenanthrene	85-01-8	1.9%
Fluoranthene	206-44-0	0.6%
9H-fluorene-9-one	486-25-9	0.4%

2-phenylnaphthalene	35465-71-5	0.2%
Sum		5.2%

#### Remarks:

The given percentage of the total peak area indicates the part of the compound relative to the total area of the chromatogram. This is a qualitative information regarding the main compounds of the mixture. It isn't possible to transform this information into a quantitative measure, as the analysator is not calibrated for the individual compounds. Therefore the two different adsorbents of the same sample cannot be compared, as the total area of the two samples maybe completely different.

## 9.7. Measurements of dioxins and furans

The concentrations of dioxins und furans are expressed in toxicity equivalents (I-TEQ) according to the International Toxicity Equivalent Factors (I-TEF, see appendix 5).

The measurement shows a range of values, as some or all of the quantified isomeres lie below the detection limit. The lower number is the sum of all isomeres which could be quantified (above detection limit), the higher number includes also the detection limits of all other isomeres as an upper limit.

## a) Measurement between rapid oxidation chamber and quenche

#### Measured concentration

Sampling interval		V	m (ng I-TEQ)		C <sub>n</sub> *
from	til	Nm³	Min.	Max.	ng I-TEQ/m³
14:35	20:35	10.625	0.00	0.51	< 0.02

#### Calculated mass flow

ſ	PCDD/F
	μg I-TEQ/h
	not available

<sup>\*</sup> the concentration is expressed at the oxygen-content at the sampling point

#### b) Measurement at the exhaust line

## Measured concentration

Sampling interval		V	m (ng I-TEQ)		C <sub>n</sub> *
from	til	Nm³	Min.	Max.	ng I-TEQ/m³
14:40	20:20	12.535	1.7	3.2	0.25 ± 0.11

<sup>\*</sup> the concentration is corrected for the dilution-air effect

#### Calculated mass flow

PCDD/F			
μg I-TEQ/h			
0.08 ± 0.03			

## 10. Results of test HD-3 (18.1.2001)

#### Note:

All concentrations refer to dry gas at normal conditions (273 K, 101.3 kPa) and are normalised to the oxygencontent of the gas before addition of dilution-air.

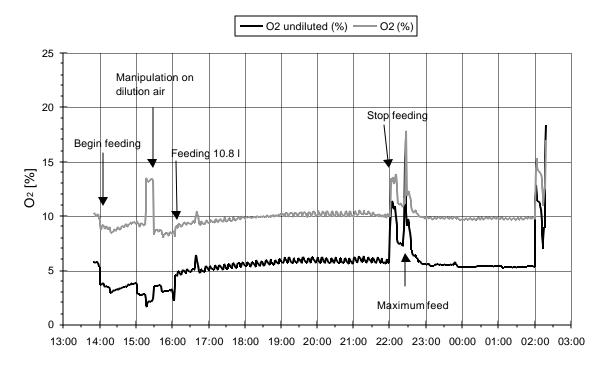
## 10.1. Measurements of O<sub>2</sub>, CO<sub>2</sub>, CO, NO<sub>2</sub> and HC

## a) Comparison of oxygen-content before and after dilution-air inlet

18.01.2001		Before HEPA	Behind HEPA
Interval		O <sub>2</sub> undiluted	O <sub>2</sub> diluted
from	til	%	%
14:00	15:00	$3.5 \pm 0.2$	$9.0 \pm 0.4$
15:00	16:00	$2.9 \pm 0.2$	$9.6 \pm 0.5$
16:00	17:00	$4.9 \pm 0.2$	$9.3 \pm 0.5$

Ī	17:00	18:00	$5.3 \pm 0.3$	$9.6 \pm 0.5$
	18:00	19:00	$5.6 \pm 0.3$	$9.9 \pm 0.5$
	19:00	20:00	$5.9 \pm 0.3$	$10.2 \pm 0.5$
	20:00	21:00	$5.9 \pm 0.3$	$10.2 \pm 0.5$
	21:00	22:00	$5.9 \pm 0.3$	10.1 ± 0.5
	22:00	23:00	$8.0 \pm 0.4$	$11.4 \pm 0.6$
	23:00	00:00	$5.5 \pm 0.3$	$9.8 \pm 0.5$
	00:00	01:00	$5.3 \pm 0.3$	$9.7 \pm 0.5$
	01:00	02:00	$5.3 \pm 0.3$	$9.7 \pm 0.5$
	14:00	02:00	$5.3 \pm 0.3$	$9.9 \pm 0.5$

Grafic representation of the oxygen-content before and after dilution (time-base one minute)



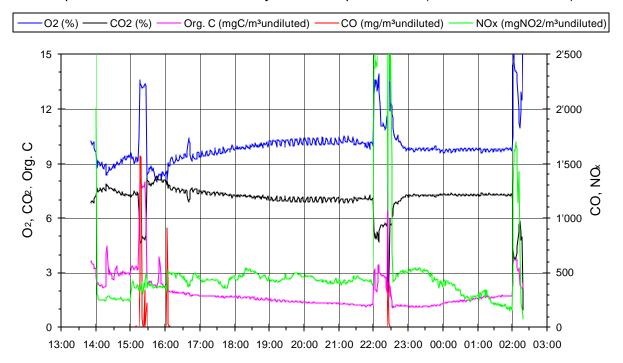
## b) Continuously measured concentrations in the exhaust line

Interval		O <sub>2</sub>	CO <sub>2</sub>	CO *	NOx *	Org. C *
from	til	%	%	mg/m³	$mgNO_2/m^3$	mgC/m³
14:00	15:00	$9.0 \pm 0.4$	$7.5 \pm 0.4$	<9	$263 \pm 30$	3 ± 1
15:00	16:00	$9.6 \pm 0.5$	$7.2 \pm 0.4$	$59 \pm 10$	$380 \pm 41$	3 ± 1
16:00	17:00	$9.3 \pm 0.5$	$7.5 \pm 0.4$	$34 \pm 9$	$458 \pm 49$	2 ± 1
17:00	18:00	$9.6 \pm 0.5$	$7.3 \pm 0.4$	<9	$422 \pm 46$	2 ± 1
18:00	19:00	$9.9 \pm 0.5$	$7.1 \pm 0.4$	<9	$482 \pm 53$	2 ± 1
19:00	20:00	10.2 ± 0.5	$7.0 \pm 0.4$	<9	$452 \pm 50$	1 ± 1
20:00	21:00	10.2 ± 0.5	$7.0 \pm 0.4$	<9	$432 \pm 48$	1 ± 1
21:00	22:00	10.1 ± 0.5	$7.0 \pm 0.4$	<9	$427 \pm 47$	1 ± 1
22:00	23:00	11.4 ± 0.6	$6.0 \pm 0.3$	14 ± 8	1'639 ± 191	2 ± 1
23:00	00:00	$9.8 \pm 0.5$	$7.2 \pm 0.4$	<9	$487 \pm 53$	1 ± 1
00:00	01:00	9.7 ± 0.5	$7.3 \pm 0.4$	<9	$333 \pm 36$	1 ± 1
01:00	02:00	$9.7 \pm 0.5$	$7.3 \pm 0.4$	<9	$235 \pm 29$	2 ± 1

|--|

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

Grafic representation of the continuously measured parameters (time-base one minute)



## 11. Measuring methods

The measurements are performed according to the EPA recommendations, and if not available or applicable according to the European (CEN guidelines) or German (VDI guidelines). All these sampling methods are validated methods. The analyses of the materials was performed by Labor Dr. Meyer AG in Berne, an accredited laboratory according to EN45'001. Validated methods of the laboratory are labelled VS-XXX. If the laboratory followed NIOSH or EPA methods these are validated through these bodies.

### Volumetric flowrate:

Measurement of flue-gas velocity with a Prandtl's-tube and an electronic micro-differential-pressure-gauge.

Detection limit: 170 Nm $^3$ /h Uncertainity of measurement at this low flow:  $\pm$  20 %<sub>rel</sub>

### Fluegas temperature:

Measurement by a NiCr/Ni-thermocouple.

Measuring range: -50 to 200° C Uncertainity of measurement: ± 3°C

### **Humidity of fluegas:**

### Method A:

The humidity of the fluegas is determined by condensation in a reflux condenser during the measurement of particulate matter or by determination of the condensate in the sampling apparatus for DMMP respectively dioxins and furans. The remaining humidity of the gas behind the condenser is equal to the saturation vapour pressure at the temperature of the condenser and is taken into account when calculating the humidity of the fluegas.

### Method B:

The humidity of the fluegas is determined by determination of the condensate in the impingers used to sample aldehydes. The remaining humidity of the gas behind the impingers is equal to the saturation vapour pressure at the temperature of the impingers and is taken into account when calculating the humidity of the fluegas.

Detection limit: 0.01 kg/Nm<sup>3</sup> Uncertainity of measurement: ± 10 %<sub>rel</sub>

### Concentration of hydrocyanic acid:

The samples are taken according to NIOSH method no 7904 by absorption of hydrogen cyanide in 0.1 NaOH in deionised water The sample is taken in a split of the flow behind the particulate filter when sampling particulates. The gases pass a titanium probe, a heated quarz plane filter, a heated PTFE tube (3 m length) and two gas wash bottles in series. The gas volume is measured by a dry gas counter behind a silica gel drier.

The analysis of the solutions for HCN was performed by photometry (Labor Dr. Meyer AG, method VS-143).

Detection limits (derived from solution blanks): 1.5 mg/m<sup>3</sup>

Uncertainity of measurement: ± 10 %rel

### Concentration of hydrochloric acid:

The samples are taken according to EPA method no 0026 by absorption of hydrogen chloride in 0.1 N sulfuric acid in deionised water. The sample is taken in a split of the flow behind the particulate filter when sampling particulates. The gases pass a titanium probe, a heated quarz plane filter, a heated PTFE tube (3 m length) and two gas wash bottles in series. The gas volume is measured by a dry gas counter behind a silica gel drier.

The analysis of the solutions for Cl<sup>-</sup> was performed by ionchromatography (Labor Dr. Meyer AG, method VS-124).

Detection limits (derived from solution blanks): 0.05 mg/m<sup>3</sup>

Uncertainty of measurement: the larger of  $\pm$  0.1 mg/m<sup>3</sup> and  $\pm$  10 %rel

### **Concentration of aldehydes:**

The samples are taken according to EPA method no 0011 by absorption of aldehydes in 2,3-dinitrophenylhydrazine. The gases are aspired isocinetically through a heated titanium probe (120  $\pm$  14°C) and three impingers in series. The gas volume is measured by a gas counter after a silica gel drier.

The analysis of the solutions for formaldehyde and acetaldehyde was performed by high-pressure liquid chromatography according to EPA method no 8315A.

Detection limits (derived from solution blanks):

- aldehydes: 0.05 mg/m<sup>3</sup>

Uncertainity of measurement (overall, including uncertainity of oxygen measurement):

- aldehydes: the larger of  $\pm$  0.1 mg/m<sup>3</sup> and  $\pm$  20 %rel

### Concentration of monoethynolamine (MEA):

The samples are taken according to EPA method no 2007 by adsorption on silicagel. The gases pass a glass probe and two beds of silicagel in series. The gas volume is measured by a gas counter after a silica gel drier.

The analysis of the two adsorption beds was performed after derivatisation by GC-MS.

Detection limits (derived from adsorbent blanks): 0.2 mg/m<sup>3</sup>

Uncertainity of measurement: ± 10 %rel

### Concentration of dimethyl-methylphosphonate (DMMP):

The samples are taken using a LAGA-train (see sampling of dioxins and furans). This is possible, as DMMP is very well sater soluble and therefore retained in the condensate behind the intense cooler. The gas volume is measured by a gas counter after a silica gel drier.

The analysis of the solution for DMMP was performed by GC-MS.

Detection limits (derived from solution blanks): 0.5 mg/m<sup>3</sup>

Uncertainity of measurement: not investigated, assumed ± 30 %rel

### Particulate matter (Dust):

The samples were taken according to VDI method no. 2066, sheet 1 (VDI: Association of German engineers; the method corresponds to EPA method no 5).

The particulates are extracted isokinetically by a heated probe and filtered through a heated, flat quarz-filter (both at  $120 \pm 14^{\circ}$ C) situated outside the flue-gas duct. The volumetric flow-rate passing the filter is monitored by a flowmeter. The water-vapour in the flue-gas is condensated in a reflux condenser situated between filter and gas-pump. The total dry volume of gas extracted from the flue-gas is measured by a positive displacement gas meter.

The amount of particulates collected on the filter is determined gravimetrically.

Detection limit: 0.2 mg/m<sup>3</sup>

Uncertainity of measurement: the larger of  $\pm$  0.5 mg/m<sup>3</sup> and  $\pm$  10 %<sub>rel</sub>

### Trace metals:

The samples were taken according to VDI method no. 3868, sheet 1 (VDI: Association of German engineers; the method corresponds to EPA method no 29).

The quarz-filter used to determine particulates is also used for the analysis of particulate trace metals. In addition the filter-passing part of the trace metals is determined by taking a sample in a split of the flow behind the particulate filter when sampling particulates. The gases pass a titanium probe, a heated quarz plane filter (sampling particulates), a heated PTFE tube (3 m length) and three gas wash bottles in series. The volumetric flow-rate passing the bubblers is monitored by a flowmeter. The total dry volume of gas passing the impingers is measured by a positive displacement gas meter.

The gaseous trace metals are collected in three bubblers containing 5 % nitric acid and 10 % peroxide. The trace metal concentrations in the digested filters (HF + microwave with HNO<sub>3</sub>) and the absorption solutions are determined by ICP-AES (Labor Dr. Meyer AG, method VS-157).

### **Polychlorinated Dioxins and Furans:**

The samples were taken according to VDI method no. 3499 (VDI: Association of German engineers; the method corresponds to EPA method no 23).

The sample is extracted isokinetically by a glass lined titanium probe and filtered through a quarz fiber filter spiked with 1,2,3,4-TCDD situated outside the flue gas duct, shock-cooled to below 10°C in a condenser and adsorbed in an adsorbent trap filled with XAD-2. The volumetric flow-rate passing the assembly is monitored by a flowmeter. The total dry volume of gas extracted from the flue gas is measured by a positive displacement gas meter.

The dioxins and furans are extracted following EPA method 23. Before cleaning procedures begin, <sup>13</sup>C-labeled standards (7 for dioxins, 10 for furans) of all 2,3,7,8-substituted isomeres are added. The extract is injected into a high resolution gas chromatographer HP 5890 series II with mass selective detector HP 5971 A in single ion mode (Labor Dr. Meyer AG, method VS-122).

### Identification of volatile and semi-volatile organic compounds (VOC's/SVOC's):

The samples are taken by adsorption on two different adsorbents; Carbopack B for semi-volatile VOC's (6 to 22 carbon atoms) and Carboxen for volatile VOC's (2 to 5 carbon atoms). The gases pass a short glass probe, a glass condensate trap and the adsorbent tubes. The gas volume is measured by a gas counter after a silica gel drier (flow rate 0.1 l/min).

The analysis of the VOC's is performed by thermo-desorption and GC-MS, using the Whiley library (150'000 spectra, ca. 80'000 compounds).

Detection limits (estimated; depending on compound): typically some μg/m<sup>3</sup>.

### Sample gas conditioning for continuous measurements:

### Measurement before dilution air inlet:

Sampling occurs through a stainless-steel tube in the flue gas, followed by a heated ceramic filter (150°C). The dust free gases pass an unheated PVC-tube (30 m length) and are dried in a condensing gas cooler. The dry gas is pumped into the oxygen-analyser.

### Measurement in exhaust line:

Sampling occurs through a glass tube in the flue gas, followed by a heated quarz filter (150°C). The dust free gases pass a heated PTFE tube (20 m length) and a heated pump, and are dried in a condensing gas cooler. The dry gas is distributed to the individual instruments by means of a heated distributor, except for the dew point (humidity) and the FID (volatile organic compounts) instruments, where the sample gas is branched off before the gas drier by heated PTFE tubes.

### Oxygen (O<sub>2</sub>):

Continuous measurement with an analyser type Oxynos 100 by Rosemount. Measuring principle: magnetic susceptibility.

Measuring range: 0 - 30 %<sub>Vol.</sub>

Calibration by ambient air; 20.95 %<sub>Vol.</sub>. Zero point adjustment by pure nitrogen.

Uncertainity of measurement: the larger of  $\pm$  0.2 %<sub>Vol.</sub> and  $\pm$  3 %<sub>rel.</sub>

## Carbon monoxide (CO)- and carbon dioxide (CO<sub>2</sub>):

Continuous measurement with an analyser type BINOS 1000 by Rosemount. Measuring principle: non-dispersive infrared absorption.

Measuring ranges used:

CO: 0 - 500 ppm<sub>Vol.</sub> CO<sub>2</sub>: 0 - 20 %<sub>Vol</sub>

Calibration:

zero point: pure nitrogen

span: CO: 39.5 ppm $_{Vol.}$  CO  $\pm$  2  $\%_{rel}$  in pure nitrogen

 $CO_2$ : 15.0 %<sub>Vol.</sub>  $CO_2 \pm 2$  %<sub>rel</sub> in pure nitrogen

Uncertainity of measurement:

CO: the larger of  $\pm$  6 mg/m<sup>3</sup> and  $\pm$  10 %<sub>rel.</sub> CO<sub>2</sub>: the larger of  $\pm$  0.2 %<sub>Vol.</sub> and  $\pm$  5 %<sub>rel.</sub>

### Nitrogen oxides (NO<sub>x</sub>):

Continuous measurement with an two channel (NO and NO<sub>x</sub>) analyser type CLD 502 by Tecan. Measuring principle: chemiluminescence.

Measuring range used: 0 - 500/1'000/5'000 ppm<sub>Vol.</sub>

Calibration by pure nitrogen (zero point) and 446 ppm<sub>Vol.</sub> NO  $\pm$  2 %<sub>rel</sub> in pure nitrogen (span) Uncertainity of measurement: the larger of  $\pm$  10 mg/m<sup>3</sup> and  $\pm$  10 %<sub>rel</sub>

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Interferences due to chemiluminescence quenching by oxygen, carbon dioxide and residual water vapour are corrected.

### Volatile organic compounds (measured by FID):

The samples were taken according to VDI method no. 3481, sheet 3 (VDI: Association of German engineers; the method corresponds to EPA method no 25).

The measurement of volatile organic compounds is carried out using an FID (flame ionisation detector), type JUM 3-100 by Kull. The apparatus is calibrated by a propane ( $C_3H_8$ ) calibration gas. The results are converted into total carbon (Org. C) units.

Measuring range used: 0 - 100 ppm<sub>Vol.</sub>

Calibration by pure nitrogen (zero point) and 8.8 ppm<sub>Vol.</sub>  $C_3H_8 \pm 2$  %<sub>rel</sub> in pure nitrogen (span) Uncertainty of measurement: the larger of  $\pm$  0.2 mg/m<sup>3</sup> and  $\pm$  20 %<sub>rel</sub>

### Data acquisition:

The data of the continuous analysers are recorded digitally by a PC based data acquisition system.

Gerlafingen, 28. March 2002

Dr. Graf AG Umweltschutz und Wärmetechnik 4563 Gerlafingen

Dr. M. Andrée

H.U. Bieri

## **Appendix**

### Explanation of the abbreviations used:

- T flue gas temperature
- b barometric pressure (ambient pressure)
- Δp pressure difference between ambient pressure and the pressure inside the gas duct (negativ = underpressure)
- f humidity of gas normalized to standard contitions (0°C, 1013 mbar), dry gas
- v gas velocity
- V<sub>b</sub> volumetric flow rate at operating conditions
- V<sub>n,f</sub> volumetric flow rate normalized to standard contitions (0°C, 1013 mbar), wet gas
- $V_{n,tr}^{'}$  volumetric flow rate normalized to standard contitions (0°C, 1013 mbar), dry gas
- V total volume of gas used for grab sample, normalized to standard conditions (0°C, 1013 mbar), dry gas
- m mass of particulates on filter / mass absorbed in solution / mass in sampling train
- c<sub>n</sub> concentration of compound normalzed to standard conditions (0°C, 1013 mbar), dry gas

## Appendix 1: Detailed results of particulates and trace metals (GB-1)

### a) Particulates

Sampling interval		V	m	Particulates *	Humidity	O <sub>2</sub>
from til		Nm³	mg	mg/m³	kg/m³	%
22:00	01:00	5.844	650	170 ± 20	$0.109 \pm 0.013$	$10.9 \pm 0.5$

<sup>\*</sup> the concentration is corrected for the dilution-air effect

### b) Trace metals

Chro	mium	dust bound			pas	filter	sum	
Samplin	g interval	V	m	Cn *	V	m	C <sub>n</sub> *	Cn *
from	til	Nm³	μg	mg/m³	Nm³	μg	mg/m³	mg/m³
22:00	01:00	5.844	3'800	0.99	0.277	0.1	< 0.001	0.99

Cop	per	dust bound			pas	filter	sum	
Sampling	g interval	V	m	C <sub>n</sub> *	V	m	Cn *	Cn *
from	til	Nm³	μg	mg/m³	Nm³	μg	mg/m³	mg/m³
22:00	01:00	5.844	350	0.093	0.277	0.2	< 0.001	0.094

Iro	on	dust bound			pas	ilter	sum	
Sampling	g interval	V	m	C <sub>n</sub> *	V	m	Cn *	Cn *
from	til	Nm³	μg	mg/m³	Nm³	μg	mg/m³	mg/m³
22:00	01:00	5.844	300	0.078	0.277	2.3	0.013	0.090

	Zi	nc	dust bound			pas	ilter	sum	
S	Sampling	g interval	V	m	c <sub>n</sub> *	V	m	Cn *	Cn *
f	from	til	Nm³	μg	mg/m³	Nm³	μg	mg/m³	mg/m³
2	22:00	01:00	5.844	320	0.083	0.277	2.0	0.011	0.094

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

## Appendix 2: Detailed results of aldehydes, HCN and MEA (GB-1)

### a) Aldehydes

behind HEPA, 12.01.01		formaldehyde		acet	aldehyde	sum	O <sub>2</sub>	
Samplin	ng interval	V	m	C <sub>n</sub> *	m	C <sub>n</sub> *	Cn *	
from	til	Nm³	mg	mg/m³	mg	mg/m³	mg/m³	%
20:15	21:15	0.896	0.18	$0.33 \pm 0.07$	0.22	$0.41 \pm 0.08$	$0.74 \pm 0.11$	10.9 ± 0.5

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

### b) Hydrocyanic acid

Sampling	g interval	V	m	Cn *	O <sub>2</sub>
from til		Nm³	mg	mg/m³	%
22:00 01:00		0.403	< 0.4	< 1.6	10.9 ± 0.5

<sup>\*</sup> the concentration is corrected for the dilution-air effect

### c) Monoethanoleamine

Sampling	g interval	V	m	C <sub>n</sub> *	O <sub>2</sub>
from	from til		mg	mg/m³	%
22:20	00:20	0.020	< 0.002	< 0.2	11.1 ± 0.6

<sup>\*</sup> the concentration is corrected for the dilution-air effect

## Appendix 3: Detailed results of particulates and trace metals (HD-2)

### a) Particulates

Sampling	g interval	V	m	Particulates *	Humidity	O <sub>2</sub>
from til		Nm³	mg	mg/m³	kg/m³	%
16:45	19:30	5.212	1'100	270 ± 30	0.10 ± 0.01	9.1 ± 0.5

<sup>\*</sup> the concentration is corrected for the dilution-air effect

### b) Trace metals

Chro	mium	dust bound			pa	ilter	sum	
Samplin	g interval	V	m	Cn *	V	m	Cn *	Cn *
from	til	Nm³	μg	mg/m³	Nm³	μg	mg/m³	mg/m³
16:45	19:30	5.212	1'400	0.36	0.248	< 0.1	< 0.001	0.36

Cop	per	dust bound			pas	ilter	sum	
Samplin	g interval	V	m	Cn *	V	m	Cn *	Cn *
from	til	Nm³	μg	mg/m³	Nm³	μg	mg/m³	mg/m³
16:45	19:30	5.212	2'500	0.62	0.248	0.5	0.003	0.62

Iro	on	dust bound			pas	ilter	sum	
Sampling	g interval	V	m	C <sub>n</sub> *	V	m	Cn *	C <sub>n</sub> *
from	til	Nm³	μg	mg/m³	Nm³	μg	mg/m³	mg/m³
16:45	19:30	5.212	520	0.13	0.248	1.3	0.007	0.14

Zi	nc	dust bound			pas	ilter	sum	
Sampling	g interval	V	m	Cn *	V	m	Cn *	Cn *
from	til	Nm³	μg	mg/m³	Nm³	μg	mg/m³	mg/m³
16:45	19:30	5.212	12'000	2.9	0.248	1.3	0.007	2.9

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

## Appendix 4: Detailed results of aldehydes, HCI and HCN (HD-2)

### a) Aldehydes

behind	HEPA, 1	2.01.01	formaldehyde		acetaldehyde		sum	O <sub>2</sub>
Samplin	ng interval	V	m	C <sub>n</sub> *	m	C <sub>n</sub> *	Cn *	
from	til	Nm³	mg	mg/m³	mg	mg/m³	mg/m³	%
15:30	16:30	1.166	0.20	0.21 ± 0.04	0.16	$0.17 \pm 0.03$	$0.38 \pm 0.06$	$9.2 \pm 0.5$

<sup>\*</sup> the concentrations are corrected for the dilution-air effect

### b) Hydrochloric acid

Sampling	Sampling interval		m	C <sub>n</sub> *	O <sub>2</sub>
from	til	Nm³	mg	mg/m³	%
16:45	19:30	0.336	0.09	$0.36 \pm 0.04$	9.1 ± 0.5

<sup>\*</sup> the concentration is corrected for the dilution-air effect

### c) Hydrocyanic acid

Sampling	Sampling interval		m	C <sub>n</sub> *	O <sub>2</sub>
from	til	Nm³	mg	mg/m³	%
16:45	19:30	0.286	< 0.4	< 2	9.1 ± 0.5

<sup>\*</sup> the concentration is corrected for the dilution-air effect

## Appendix 5: Detailed results of dioxins and furans analyses (HD-2)

The following table shows the absolute amounts of the isomeres in the analysed sample, the toxicity factors according to International Toxicity Equivalent Factors (I-TEF) and the toxicity equivalents calculated for the sample (I-TEQ).

In the following table the absolute amounts of the analysed sample and the toxicity equivalents (multiplied with the Toxicity Equivalent Factors (I-TEF)) are listed.

### a) Measurement between rapid oxidation chamber and quenche

Dioxin	Content ng abs.	Toxicity Equivalent Factors I-TEF	ng I-TEQ absolute
2,3,7,8-TCDD	<0.12	1	<0.120
Sum Tetrachlordibenzo-p-dioxin	0.61		
1,2,3,7,8-PeCDD	<0.20	0.5	<0.100
Sum Pentachlordibenzo-p-dioxin	6.39		
1,2,3,4,7,8-HxCDD	<0.34	0.1	<0.034
1,2,3,6,7,8-HxCDD	<0.31	0.1	<0.031
1,2,3,7,8,9-HxCDD	<0.38	0.1	<0.038
Sum Hexachlordibenzo-p-dioxin	<3.41		
1,2,3,4,6,7,8-HpCDD	<0.89	0.01	<0.009
Sum Heptachlordibenzo-p-dioxin	<1.77		
Octachlordibenzo-p-dioxin	<4.00	0.001	<0.004

Furan	Content ng abs.	Toxicity Equivalent Factors I-TEF	ng I-TEQ absolute
2,3,7,8-TCDF	<0.13	0.1	<0.013
Sum Tetrachlordibenzofuran	2.01		
1,2,3,7,8-PeCDF	<0.12	0.05	<0.006
2,3,4,7,8-PeCDF	<0.06	0.5	< 0.030
Sum Pentachlordibenzofuran	0.81		
1,2,3,4,7,8-HxCDF	<0.23	0.1	<0.023
1,2,3,6,7,8-HxCDF	<0.26	0.1	<0.026
1,2,3,7,8,9-HxCDF	<0.31	0.1	<0.031
2,3,4,6,7,8-HxCDF	<0.39	0.1	< 0.039
Sum Hexachlordibenzofuran	<2.95		
1,2,3,4,6,7,8-HpCDF	<0.30	0.01	<0.003
1,2,3,4,7,8,9-HpCDF	<0.58	0.01	<0.006
Sum Heptachlordibenzofuran	<1.75		
Octachlordibenzofuran	<2.56	0.001	<0.003

The sum includes the 2,3,7,8-substituted isomeres.

The total toxicity equivalent in the sample is 0.52 ng TEQ abs. at the maximum.

### b) Measurement at the exhaust line

Dioxin	Content ng abs.	Toxicity Equivalent Factors I-TEF	ng I-TEQ absolute
2,3,7,8-TCDD	<0.23	1	<0.230
Sum Tetrachlordibenzo-p-dioxin	23		
1,2,3,7,8-PeCDD	<1.3	0.5	<0.630
Sum Pentachlordibenzo-p-dioxin	5.9		
1,2,3,4,7,8-HxCDD	<0.45	0.1	<0.045
1,2,3,6,7,8-HxCDD	<0.40	0.1	<0.040
1,2,3,7,8,9-HxCDD	<0.44	0.1	<0.044
Sum Hexachlordibenzo-p-dioxin	1.9		
1,2,3,4,6,7,8-HpCDD	<2.5	0.01	<0.025
Sum Heptachlordibenzo-p-dioxin	<5.0		
Octachlordibenzo-p-dioxin	<3.2	0.001	<0.003

Furan	Content ng abs.	Toxicity Equivalent Factors I-TEF	ng I-TEQ absolute
2,3,7,8-TCDF	8.8	0.1	0.879
Sum Tetrachlordibenzofuran	328		
1,2,3,7,8-PeCDF	1.4	0.05	0.070
2,3,4,7,8-PeCDF	1.1	0.5	0.565
Sum Pentachlordibenzofuran	21		
1,2,3,4,7,8-HxCDF	1.8	0.1	0.182
1,2,3,6,7,8-HxCDF	<1.5	0.1	<0.151
1,2,3,7,8,9-HxCDF	<1.2	0.1	<0.119
2,3,4,6,7,8-HxCDF	<1.3	0.1	<0.125
Sum Hexachlordibenzofuran	11		
1,2,3,4,6,7,8-HpCDF	<4.5	0.01	<0.045
1,2,3,4,7,8,9-HpCDF	<0.81	0.01	<0.008
Sum Heptachlordibenzofuran	< 10.6		
Octachlordibenzofuran	<3.2	0.001	<0.003

The sum includes the 2,3,7,8-substituted isomeres.

The total toxicity equivalent in the sample is at the minimum 1.70 ng TEQ abs. and 3.16 ng TEQ abs.at the maximum.

## MGC-Plasma AG, Muttenz

## Emission Measurements During Demonstration Tests of the PLASMOX System Additional Analyses

Report no. : 92.3949-Z

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April 2001

### 1. Introduction

As ordered by MGC-Plasma Ltd, Muttenz, Switzerland, the following additional analyses have been performed on samples of the demonstration test HD-2:

## Analysed parameters for stack testing (behind ventilator) Grab sampling during test HD-2 (17.1.2001)

- Phosphorus (particle bound and volatile parts, one sample, 3 h sampling time)
- Aluminium (particle bound part only, one sample, 3 h sampling time)

Originally, phosphorus was supposed to be analysed on the samples from test-run GB-1; unfortunately the filter of this run had been consumed by former analyses.

### 2. Customer

MGC-Plasma AG Mr. H. Felix Hofackerstrasse 24 4132 Muttenz

## 3. Plant

See report no. 92.3949.

## 4. Accomplishment of Measurements

See report no. 92.3949.

### Measuring points at the exhaust line

Grab sampling of particulates, phosphorus and aluminium:

1 vertically oriented EMPA-connection piece in slightly tilted PVC-pipe before exhaust.

### **Operating conditions**

The operating conditions were registered by the operators of MGC-Plasma Ltd.

## 5. Additional results of test HD-2 (17.1.2001)

### Note:

All concentrations refer to dry gas at normal conditions (273 K, 101.3 kPa) and are normalised to the oxygen content of the gas before addition of dilution-air as measured for the same interval of time.

### 5.1. Measurements of the volumetric flow rate

Plant: PLASMOX system, sampling point: exhaust line Cross section at sampling point: Ø 250 mm, Area: 0.049 m²

Time	Т	b	Δр	f*	٧	$V_b$	$V_{n,f}$	$V_{n,tr}$
	°C	mbar	mbar	kg/m³	m/s	m³/h	Nm³/h	Nm³/h
14:05	57	985	0.1	0.11	3.5	610	490	440 ± 70

<sup>\*</sup> average humidity during the dioxine- and furane-measurements

### Remark:

The volumetric flow rate is kept stable by the system. A change in the flow rate at the exhaust is only possible, if the amount of dilution air is manually changed.

### 5.2. Measurements of particulates, phosphorus and aluminium

### Measured concentrations

Samplin	ng interval	Particulates	Phosphorus	Aluminium
from	til	mg/m³	mg/m³	mg/m³
16:45	19:30	270 ± 30	2.2 ± 0.4	0.05 ± 0.01

the concentrations are normalised to the oxygen content of the gas before addition of dilution-air

### Calculated mass flows

Samplin	g interval	Particulates	Phosphorus	Aluminium	
from	from til		g/h	g/h	
16:45	16:45 19:30 90		0.8 ± 0.2	0.017 ± 0.004	

The partition of phosphorus into a dust-bound and a filter-passing part is shown in the detailed results in appendix 1.

## 6. Measuring methods

The measurements are performed according to the EPA recommendations, and if not available or applicable, according to the European (CEN) or German (VDI) guidelines. All these sampling methods are validated methods. The analyses of the materials was performed by Labor Dr. Meyer AG in Berne, an accredited laboratory according to EN45'001. Validated methods of the laboratory are labelled VS-XXX. If the laboratory followed NIOSH or EPA methods these are validated through these bodies.

### Volumetric flowrate:

Measurement of flue-gas velocity with a Prandtl's-tube and an electronic micro-differential-pressure-gauge.

Detection limit: 170 Nm $^3$ /h Uncertainity of measurement at this low flow:  $\pm$  20  $\%_{rel}$ 

### Fluegas temperature:

Measurement by a NiCr/Ni-thermocouple. Measuring range: -50 to 200° C Uncertainity of measurement: ± 3°C

### Particulate matter (Dust):

The samples were taken according to VDI method no. 2066, sheet 1 (VDI: Association of German engineers; the method corresponds to EPA method no 5).

The particulates are extracted isokinetically by a heated probe and filtered through a heated, flat quarz-filter (both at  $120 \pm 14^{\circ}\text{C}$ ) situated outside the flue-gas duct. The volumetric flow-rate passing the filter is monitored by a flowmeter. The water-vapour in the flue-gas is condensated in a reflux condenser situated between filter and gas-pump. The total dry volume of gas extracted from the flue-gas is measured by a positive displacement gas meter.

The amount of particulates collected on the filter is determined gravimetrically.

Detection limit: 0.2 mg/m<sup>3</sup>

Uncertainity of measurement: the larger of  $\pm$  0.5 mg/m<sup>3</sup> and  $\pm$  10 %<sub>rel</sub>

### Phosphourous, Aluminium:

The samples were taken according to VDI method no. 3868, sheet 1 (VDI: Association of German engineers; the method corresponds to EPA method no 29).

The quarz-filter used to determine particulates is also used for the analysis of particulate trace metals. In addition the filter-passing part of the trace metals is determined by taking a sample in a split of the flow behind the particulate filter while sampling particulates. The gases pass a titanium probe, a heated quarz plane filter (sampling particulates), a heated PTFE tube (3 m length) and three gas wash bottles in series. The volumetric flow-rate passing the bubblers is monitored by a flowmeter. The total dry volume of gas passing the impingers is measured by a positive displacement gas meter.

The gaseous trace metals are collected in three bubblers containing 5 % nitric acid and 10 % peroxide. The trace metal concentrations in the digested filters (HF + microwave with HNO<sub>3</sub>) and the absorption solutions are determined by ICP-AES (Labor Dr. Meyer AG, method VS-132 for phosphorus and VS-157 for aluminium).

Gerlafingen, 25 April 2001

Dr. Graf AG Umweltschutz und Wärmetechnik 4563 Gerlafingen

Dr. M. Andrée

H.U. Bieri

## **Appendix**

### Explanation of the abbreviations used:

- T flue gas temperature
- b barometric pressure (ambient pressure)
- Δp pressure difference between ambient pressure and the pressure inside the gas duct (negativ = underpressure)
- f humidity of gas normalized to standard contitions (0°C, 1013 mbar), dry gas
- v gas velocity
- V<sub>b</sub> volumetric flow rate at operating conditions
- V<sub>n,f</sub> volumetric flow rate normalized to standard contitions (0°C, 1013 mbar), wet gas
- V<sub>n tr</sub> volumetric flow rate normalized to standard contitions (0°C, 1013 mbar), dry gas
- V total volume of gas used for grab sample, normalized to standard conditions (0°C, 1013 mbar), dry gas m mass of particulates on filter / mass absorbed in solution / mass in sampling train
- c<sub>n</sub> concentration of compound normalzed to standard conditions (0°C, 1013 mbar), dry gas

## Appendix 1: Detailed results of particulates and phosphorus

### a) Particulates

Sampling interval		V	m	Particulates *	Humidity	O <sub>2</sub>
from til		Nm³	mg	mg/m³	kg/m³	%
16:45	19:30	5.212	1'100	270 ± 30	0.10 ± 0.01	9.1 ± 0.5

<sup>\*</sup> the concentration is normalised to the oxygen content of the gas before addition of dilution-air

### b) Phosphorus

Phosp	sphorus dust bound			pas	ilter	sum		
Sampling interval V		m	Cn *	$\star$ V m $c_n \star$		C <sub>n</sub> *	Cn *	
from	til	Nm³	μg	mg/m³	Nm³	μg	mg/m³	mg/m³
16:45	19:30	5.212	8'750	2.2	0.248	<12	<0.06	2.2

<sup>\*</sup> the concentrations are normalised to the oxygen content of the gas before addition of dilution-air

MGC order references : Lieferschein 344

Kostenstelle 83350

		laboratory no. :	Q3771	Q3772	Q3773
	M	GC sample designation:	GB1 before test scrubber 1	GB1 after Test scrubber 1	GB1 after test scrubber 2
Parameter	Unit	Method			
Chloride	[mg/l]	VS-124 (IC)	20.5	81.1	<0.08
Cyanide	[mg/l]	VS-143	<0.004	<0.004	0.008
Nitrate	[mg/l]	VS-124 (IC)	4.21	58.8	<0.36
Phosphorus	[mgP/l]	VS-141	0.05	34.5	7.00
Formaldehyde	[mg/l]	EPA 8315A (HPLC)	0.838	0.167	0.050
Acetaldehyde	[mg/l]	EPA 8315A (HPLC)	0.053	< 0.03	0.063
TOC	[mg/l]	VS-118	10.7	11.9	17.8
PAH**	[µg/l]	VS-103b	0.31	0.69	0.21
MEA	[mg/l]	NIOSH 2007	to be measured	to be measured	to be measured
DMMP	[mg/l]	(*)	< 0.5	< 0.5	< 0.5
Dioxins + Furans***	[ng/l]	VS-122	n.m.	n.m.	n.m.

n.m.: not measured

this method is not included in our accreditation programme
 sum of 16 compounds, detailed results see appendix PAH I+II

\*\*\* : sum of 2,3,7,8-chlorine substituted congeners of dioxins and furans, evaluation according to the International Toxicity

Equivalent Factors (ITEF); detailed results see appendix dioxins/furans 1-10

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MGC order references : Lieferschein 344

Kostenstelle 83350

		laboratory no. :	Q3774	Q3775	Q3776
	M	GC sample designation:	GB1 after test scrubber 2	GB2 after test Scrubber 1	GB2 after test Scrubber 2
Parameter	arameter Unit Method				
Chloride	[mg/l]	VS-124 (IC)	5.83	120.5	10.0
Cyanide	[mg/l]	VS-143	0.02	0.014	0.034
Nitrate	[mg/l]	VS-124 (IC)	24.5	102	57.6
Phosphorus	[mgP/l]	VS-141	25.5	60.5	61.5
Formaldehyde	[mg/l]	EPA 8315A (HPLC)	0.158	1.842	0.175
Acetaldehyde	[mg/l]	EPA 8315A (HPLC)	< 0.03	< 0.03	< 0.03
TOC	[mg/l]	VS-118	18.1	16.5	30.0
PAH**	[μg/l]	VS-103b	0.33	0.47	0.22
MEA	[mg/l]	NIOSH 2007	to be measured	to be measured	to be measured
DMMP	[mg/l]	(*)	< 0.5	< 0.5	< 0.5
Dioxins + Furans***	[ng/l]	VS-122	n.m.	n.m.	n.m.

n.m.: not measured

this method is not included in our accreditation programme
 sum of 16 compounds, detailed results see appendix PAH I+II

\*\*\* : sum of 2,3,7,8-chlorine substituted congeners of dioxins and furans, evaluation according

Equivalent Factors (ITEF); detailed results see appendix dioxins/furans 1-10

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MGC order references : Lieferschein 344

Kostenstelle 83350

		laboratory no. :	Q3777	Q3778	Q3779
	MC	GC sample designation :	GB3 after test Scrubber 1	GB3 after test Scrubber 2	HD1 before test Scrubber 1
Parameter	Unit	Method			
Chloride	[mg/l]	VS-124 (IC)	194.6	13.1	10.1
Cyanide	[mg/l]	VS-143	0.018	0.036	<0.004
Nitrate	[mg/l]	VS-124 (IC)	184	91.0	13.8
Phosphorus	[mgP/l]	VS-141	140	120	n.m.
Formaldehyde	[mg/l]	EPA 8315A (HPLC)	0.097	0.118	0.547
Acetaldehyde	[mg/l]	EPA 8315A (HPLC)	< 0.03	< 0.03	< 0.03
TOC	[mg/l]	VS-118	20.1	27.2	5.0
PAH**	[μg/l]	VS-103b	0.36	0.68	0.48
MEA	[mg/l]	NIOSH 2007	to be measured	to be measured	to be measured
DMMP	[mg/l]	(*)	< 0.5	< 0.5	n.m.
Dioxins + Furans***	[ng/l]	VS-122	n.m.	n.m.	9.65 - 9.74

n.m.: not measured

this method is not included in our accreditation programme
 sum of 16 compounds, detailed results see appendix PAH I+II

\*\*\* : sum of 2,3,7,8-chlorine substituted congeners of dioxins and furans, evaluation according

Equivalent Factors (ITEF); detailed results see appendix dioxins/furans 1-10

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MGC order references : Lieferschein 344

Kostenstelle 83350

		laboratory no. :	Q3780	Q3781	Q3782
	M	GC sample designation:	HD1 after test Scrubber 1	HD1 before test Scrubber 2	HD1 after test Scrubber 2
Parameter	Unit	Method			
Chloride	[mg/l]	VS-124 (IC)	2560	8.44	260
Cyanide	[mg/l]	VS-143	0.008	0.014	0.008
Nitrate	[mg/l]	VS-124 (IC)	23.7	8.96	11.1
Phosphorus	[mgP/l]	VS-141	n.m.	n.m.	n.m.
Formaldehyde	[mg/l]	EPA 8315A (HPLC)	0.675	0.104	0.302
Acetaldehyde	[mg/l]	EPA 8315A (HPLC)	0.034	< 0.03	< 0.03
TOC	[mg/l]	VS-118	4.1	21.8	17.7
PAH**	[μ <b>g/</b> l]	VS-103b	0.37	0.18	0.19
MEA	[mg/l]	NIOSH 2007	to be measured	to be measured	to be measured
DMMP	[mg/l]	(*)	n.m.	n.m.	n.m.
Dioxins + Furans***	[ng/l]	VS-122	10.95 - 11.11	0.21 - 0.56	0.15 - 1.50

n.m.: not measured

this method is not included in our accreditation programme
 sum of 16 compounds, detailed results see appendix PAH I+II

\*\*\* : sum of 2,3,7,8-chlorine substituted congeners of dioxins and furans, evaluation according

Equivalent Factors (ITEF); detailed results see appendix dioxins/furans 1-10

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MGC order references : Lieferschein 344

Kostenstelle 83350

		laboratory no. :	Q3783	Q3784	Q3785
	M	GC sample designation:	HD2 after test Scrubber 1	HD2 after test Scrubber 2	HD3 after test Scrubber 1
Parameter	Unit	Method			
Chloride	[mg/l]	VS-124 (IC)	2720	578	929
Cyanide	[mg/l]	VS-143	0.01	0.024	<0.004
Nitrate	[mg/l]	VS-124 (IC)	59.2	12.1	57.7
Phosphorus	[mgP/l]	VS-141	n.m.	n.m.	n.m.
Formaldehyde	[mg/l]	EPA 8315A (HPLC)	0.350	0.064	0.196
Acetaldehyde	[mg/l]	EPA 8315A (HPLC)	< 0.03	< 0.03	< 0.03
TOC	[mg/l]	VS-118	3.0	20.8	2.0
PAH**	[μg/l]	VS-103b	< 16	0.26	< 16
MEA	[mg/l]	NIOSH 2007	to be measured	to be measured	to be measured
DMMP	[mg/l]	(*)	n.m.	n.m.	n.m.
Dioxins + Furans***	[ng/l]	VS-122	2.98 - 3.06	0.05 - 0.44	1.88 - 1.98

n.m.: not measured

this method is not included in our accreditation programme
 sum of 16 compounds, detailed results see appendix PAH I+II

\*\*\* : sum of 2,3,7,8-chlorine substituted congeners of dioxins and furans, evaluation according

Equivalent Factors (ITEF); detailed results see appendix dioxins/furans 1-10

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MGC order references : Lieferschein 344

Kostenstelle 83350

		laboratory no. :	Q3786	Q3787	Q3788
	M	GC sample designation :	HD3 after test Scrubber 2	HD4 after test Scrubber 1	HD4 after test Scrubber 2
Parameter	Unit	Method			
Chloride	[mg/l]	VS-124 (IC)	1440	525	1999
Cyanide	[mg/l]	VS-143	0.008	0.006	<0.004
Nitrate	[mg/l]	VS-124 (IC)	18.7	60.9	65.8
Phosphorus	[mgP/l]	VS-141	n.m.	n.m.	n.m.
Formaldehyde	[mg/l]	EPA 8315A (HPLC)	0.099	0.165	0.067
Acetaldehyde	[mg/l]	EPA 8315A (HPLC)	< 0.03	< 0.03	< 0.03
TOC	[mg/l]	VS-118	26.6	1.8	28.8
PAH**	[μg/l]	VS-103b	< 16	< 16	< 16
MEA	[mg/l]	NIOSH 2007	to be measured	to be measured	to be measured
DMMP	[mg/l]	(*)	n.m.	n.m.	n.m.
Dioxins + Furans***	[ng/l]	VS-122	0.00 - 1.23	1.21 - 1.39	0.01 - 0.50

n.m.: not measured

this method is not included in our accreditation programme
 sum of 16 compounds, detailed results see appendix PAH I+II

\*\*\* : sum of 2,3,7,8-chlorine substituted congeners of dioxins and furans, evaluation according

Equivalent Factors (ITEF); detailed results see appendix dioxins/furans 1-10

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MGC order references : Lieferschein 344

Kostenstelle 83350

		Lab-Nr. :	Q3789	Q3790	Q3791
	MGC	sample designation :	GB1 Slag	GB2 Slag	GB3 Slag
Parameter	Unit	Method			
Si	[weight%]**	external	50	54	59
Р	[weight%]**	external	<1	1	<1
Al	[weight%]**	external	12	8	8
Na	[weight%]**	external	2	2	3
K	[weight%]**	external	<1	1	1
Ca	[weight%]**	external	20	21	21
Mg	[weight%]**	external	14	12	7
Ва	[weight%]**	external	<1	<1	<1
Cr	[weight%]**	external	1	1	<1
Fe	[weight%]**	external	<1	<1	<1
TOC	[C%]	external	< 0.05	0.12	< 0.05
Phosphorus	[mg/kg]	VS-132	1.94	2.75	0.37
Cu total	[mg/kg]	ICP-AES	11.1	32.3	69.1
Zn total	[mg/kg]	ICP-AES	5.71	3.04	1.39
Cr total	[mg/kg]	ICP-AES	2210	1270	182
Fe total	[mg/kg]	ICP-AES	274	179	2380
TOC, TVA-leachate 24h	[mg/l]	VS-118	0.32	0.32	0.31
TOC, TVA-leachate 48h	[mg/l]	VS-118	0.10	0.28	0.19
TOC, mean according toTVA	[mg/l]	VS-118	0.21	0.30	0.25
Cu, TVA-leachate 24h	[mg/l]	ICP-AES	< 0.02	< 0.02	< 0.02
Cu, TVA-leachate 48h	[mg/l]	ICP-AES	< 0.02	< 0.02	< 0.02
Cu, mean according toTVA	[mg/l]	ICP-AES	< 0.02	< 0.02	< 0.02
Zn, TVA-leachate 24h	[mg/l]	ICP-AES	0.019	0.015	0.035
Zn, TVA-leachate 48h	[mg/l]	ICP-AES	0.011	< 0.004	0.012
Zn, mean according toTVA	[mg/l]	ICP-AES	0.015	0.010	0.024
Cr, TVA-leachate 24h	[mg/l]	ICP-AES	0.017	< 0.002	0.012
Cr, TVA-leachate 48h	[mg/l]	ICP-AES	0.008	< 0.002	< 0.002
Cr, mean according toTVA	[mg/l]	ICP-AES	0.013	< 0.002	0.007
Fe, TVA-leachate 24h	[mg/l]	ICP-AES	0.041	0.003	0.016
Fe, TVA-leachate 48h	[mg/l]	ICP-AES	0.025	< 0.001	< 0.001
Fe, mean according toTVA	[mg/l]	ICP-AES	0.033	0.002	0.009
Phosphorus, TVA-leachate 24h	[mg/l]	VS-141	< 0.02	0.02	< 0.02
Phosphorus, TVA-leachate 48h	[mg/l]	VS-141	< 0.02	< 0.02	< 0.02
Phosphorus, mean according toTVA	[mg/l]	VS-141	< 0.02	< 0.02	< 0.02

n.m.: not measured

<sup>\* :</sup> this method is not included in our accreditation programme

MGC order references : Lieferschein 344

Kostenstelle 83350

		Lab-Nr. :	Q3792	Q3793	Q3794
	MG	C sample designation :	HD1	HD2	HD3/HD4
Parameter	Unit	Method			
Si	[weight%]**	external	67	64	43
Р	[weight%]**	external	<1	<1	<1
Al	[weight%]**	external	4	7	11
Na	[weight%]**	external	2	2	<1
K	[weight%]**	external	<1	<1	<1
Ca	[weight%]**	external	22	23	36
Mg	[weight%]**	external	4	4	10
Ва	[weight%]**	external	<1	<1	<1
Cr	[weight%]**	external	<1	<1	<1
Fe	[weight%]**	external	<1	<1	<1
TOC	[C%]	external	< 0.05	< 0.05	< 0.05
Phosphorus	[mg/kg]	VS-132	n.m.	n.m.	n.m.
Cu total	[mg/kg]	ICP-AES	2.53	4.6	7.34
Zn total	[mg/kg]	ICP-AES	1.36	1.53	6.65
Cr total	[mg/kg]	ICP-AES	46.3	94.8	905
Fe total	[mg/kg]	ICP-AES	102	129	135
TOC, TVA-leachate 24h	[mg/l]	VS-118	0.48	0.25	0.22
TOC, TVA-leachate 48h	[mg/l]	VS-118	0.15	0.19	0.30
TOC, mean according toTVA	[mg/l]	VS-118	0.32	0.22	0.26
Cu, TVA-leachate 24h	[mg/l]	ICP-AES	< 0.02	< 0.02	< 0.02
Cu, TVA-leachate 48h	[mg/l]	ICP-AES	< 0.02	< 0.02	< 0.02
Cu, mean according toTVA	[mg/l]	ICP-AES	< 0.02	< 0.02	< 0.02
Zn, TVA-leachate 24h	[mg/l]	ICP-AES	0.011	0.016	0.031
Zn, TVA-leachate 48h	[mg/l]	ICP-AES	0.005	0.006	0.007
Zn, mean according toTVA	[mg/l]	ICP-AES	0.008	0.011	0.019
Cr, TVA-leachate 24h	[mg/l]	ICP-AES	< 0.002	< 0.002	0.0090
Cr, TVA-leachate 48h	[mg/l]	ICP-AES	< 0.002	< 0.002	0.0080
Cr, mean according toTVA	[mg/l]	ICP-AES	< 0.002	< 0.002	0.0085
Fe, TVA-leachate 24h	[mg/l]	ICP-AES	0.030	0.025	0.016
Fe, TVA-leachate 48h	[mg/l]	ICP-AES	0.013	< 0.001	0.006
Fe, mean according toTVA	[mg/l]	ICP-AES	0.022	0.013	0.011
Phosphorus, TVA-leachate 24h	[mg/l]	VS-141	n.m.	n.m.	n.m.
Phosphorus, TVA-leachate 48h	[mg/l]	VS-141	n.m.	n.m.	n.m.
Phosphorus, mean according toTVA	[mg/l]	VS-141	n.m.	n.m.	n.m.

Liquid-Solid.xls

n.m.: not measured

<sup>\* :</sup> this method is not included in our accreditation programme

\*\* : the masses of C and O are not taken into consideration for the total mass; the quantification of these two elements cannot be done using the present technique.

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\*\* : the masses of C and O are not taken into consideration for the total mass; the quantificati cannot be done using the present technique.

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# appendix to report Nr. 01-03-0007 PAH in scrubber liquid [μg/l]

	laboratory no. :	Q3771	Q3772	Q3773	Q3774	Q3775	Q3776	Q3777	Q3778	Q3779
1	Naphthalin	0.233	0.536	0.178	0.294	0.34	0.19	0.275	0.611	0.202
2	Acenaphthylen	0.018	0.013	0.004	0.008	<0.001	0.009	0.011	0.029	0.037
3	Acenaphthen	0.043	0.049	0.016	0.014	0.026	0.014	0.027	0.027	0.105
4	Fluoren	<0.001	0.043	<0.001	<0.001	0.026	<0.001	0.019	<0.001	0.055
5	Phenanthren	0.005	0.019	0.002	0.003	0.016	0.003	0.007	0.005	0.013
6	Anthracen	0.005	0.019	<0.001	0.005	0.002	<0.001	0.004	0.002	0.059
7	Fluoranthen	0.001	<0.001	0.001	<0.001	0.035	<0.001	0.012	0.001	0.003
8	Pyren	0.004	0.005	0.002	0.003	0.015	<0.001	0.005	0.002	0.001
9	Benz(a)anthracen	<0.001	<0.001	<0.001	<0.001	0.002	<0.001	<0.001	<0.001	<0.001
10	Chrysen	<0.001	<0.001	<0.001	<0.001	0.003	<0.001	<0.001	<0.001	<0.001
11	Benz(b)fluoranthen	0.001	<0.001	<0.001	<0.001	0.003	<0.001	<0.001	<0.001	<0.001
12	Benz(k)fluoranthen	0.001	<0.001	<0.001	<0.001	0.004	<0.001	0.001	<0.001	<0.001
13	Benz(a)pyren	<0.001	0.002	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
14	Indeno(1,2,3-cd)pyren	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
15	Dibenz(a,h)anthracen	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
16	Benz(g,h,i)perylen	<0.001	<0.001	0.006	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	sum of 16 PAH	0.311	0.686	0.209	0.327	0.472	0.216	0.361	0.677	0.475

sum PAH: only results > limit of detection

appendix PAH 1/2

# appendix to report Nr. 01-03-0007 PAH in scrubber liquid [μg/l]

	laboratory no. :	Q3780	Q3781	Q3782	Q3783*	Q3784	Q3785*	Q3786*	Q3787*	Q3788*
1	Naphthalin	0.193	0.154	0.169	< 1.0	0.209	< 1.0	< 1.0	< 1.0	< 1.0
2	Acenaphthylen	0.01	<0.001	<0.001	< 1.0	0.022	< 1.0	< 1.0	< 1.0	< 1.0
3	Acenaphthen	0.036	0.021	0.02	< 1.0	0.024	< 1.0	< 1.0	< 1.0	< 1.0
4	Fluoren	0.032	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
5	Phenanthren	0.011	0.003	0.005	< 1.0	0.004	< 1.0	< 1.0	< 1.0	< 1.0
6	Anthracen	0.088	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
7	Fluoranthen	0.002	0.001	<0.001	< 1.0	0.001	< 1.0	< 1.0	< 1.0	< 1.0
8	Pyren	<0.001	<0.001	<0.001	< 1.0	0.003	< 1.0	< 1.0	< 1.0	< 1.0
9	Benz(a)anthracen	<0.001	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
10	Chrysen	0.001	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
11	Benz(b)fluoranthen	<0.001	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
12	Benz(k)fluoranthen	<0.001	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
13	Benz(a)pyren	<0.001	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
14	Indeno(1,2,3-cd)pyren	<0.001	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
15	Dibenz(a,h)anthracen	<0.001	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
16	Benz(g,h,i)perylen	<0.001	<0.001	<0.001	< 1.0	<0.001	< 1.0	< 1.0	< 1.0	< 1.0
	sum of 16 PAH	0.373	0.179	0.194	*	0.263	*	*	*	*

sum PAH: only results > limit of detection

appendix PAH 2/2

<sup>\*</sup>difficult matrix leading to unsensitive measurement

Labor Nr.: Q3779 Probenbezeichnung: HD1 vor Test Scrubber 1

Material: Wasser Projekt Nr. : 344

Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.09	1	< 0.09
Summe-TCDD	1.50	0	
1,2,3,7,8-PCDD	1.11	0.5	0.55
Summe-PCDD	12.41	0	
123478-HxCDD	1.47	0.1	0.15
123678-HxCDD	3.07	0.1	0.31
123789-HxCDD	2.00	0.1	0.20
Summe-HxCDD	39.30	0	
1234678-HpCDD	28.92	0.01	0.29
Summe-HpCDD	67.47	0	
OCDD	30.15	0.001	0.03
2378-TCDF	0.58	0.1	0.06
Summe-TCDF	12.77	0	
12378-PCDF	1.87	0.05	0.09
23478-PCDF	5.74	0.5	2.87
Summe-PCDF	62.77	0	
123478-HxCDF	12.98	0.1	1.30
123678-HxCDF	12.17	0.1	1.22
123789-HxCDF	18.15	0.1	1.81
234678-HxCDF	0.93	0.1	0.09
Summe-HxCDF	100.18	0	
1234678-HpCDF	54.45	0.01	0.54
1234789-HpCDF	8.20	0.01	0.08
Summe-HpCDF	103.29	0	
OCDF	59.52	0.001	0.06
		-	

ı		0.00.	0.00
	Summe Tox Equivalente TE	[ ng/l TE ]	min. 9.65 max. 9.74

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

Extraktionsausbeute	2378-TCDD	13C • 80%	/_
LALI artivi i sauspeute	23/0-1000	C12.00/	0

Bern, 18. Februar 2001	LABOR DR. MEYER AG
Sachbearbeiter :	
Dr. D. Stadler, dipl. Natw. ETH	Dr. G. Meyer

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).

Labor Nr.: Q3780 Probenbezeichnung: HD1 nach Test Scrubber 1

Material: Wasser Projekt Nr. : 344

Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.16	1	< 0.16
Summe-TCDD	0.45	0	
1,2,3,7,8-PCDD	0.99	0.5	0.50
Summe-PCDD	9.38	0	
123478-HxCDD	2.13	0.1	0.21
123678-HxCDD	4.58	0.1	0.46
123789-HxCDD	2.88	0.1	0.29
Summe-HxCDD	58.06	0	
1234678-HpCDD	47.42	0.01	0.47
Summe-HpCDD	112.96	0	
OCDD	52.00	0.001	0.05
F			
2378-TCDF	0.61	0.1	0.06
Summe-TCDF	8.79	0	
12378-PCDF	1.88	0.05	0.09
23478-PCDF	4.96	0.5	2.48
Summe-PCDF	52.22	0	
123478-HxCDF	15.18	0.1	1.52
123678-HxCDF	14.10	0.1	1.41
123789-HxCDF	23.40	0.1	2.34
234678-HxCDF	1.31	0.1	0.13
Summe-HxCDF	182.51	0	
1234678-HpCDF	73.60	0.01	0.74
1234789-HpCDF	11.79	0.01	0.12
Summe-HpCDF	145.07	0	
OCDF	77.14	0.001	80.0
	Summe Tox	[ ng/l TE ]	min. 10.95
	Equivalente TE		max. 11.11

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

Extraktionsausbeute	2378-TCDD	13012	· 84%
EXII akiioi isausbeule	23/0-1600	13612	. 04/0

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).

Labor Nr.: Q3781 Probenbezeichnung: HD1 vor Test Scrubber 2

Material: Wasser Projekt Nr. : 344

Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.18	1	< 0.18
Summe-TCDD	< 1.78	0	
1,2,3,7,8-PCDD	< 0.14	0.5	< 0.07
Summe-PCDD	< 1.44	0	
123478-HxCDD	< 0.19	0.1	< 0.02
123678-HxCDD	< 0.18	0.1	< 0.02
123789-HxCDD	< 0.18	0.1	< 0.02
Summe-HxCDD	< 1.83	0	
1234678-HpCDD	0.61	0.01	0.01
Summe-HpCDD	1.55	0	
OCDD	< 3.37	0.001	< 0.00
2070 TODE	0.40	1 04 1	0.04
2378-TCDF	0.12	0.1	0.01
Summe-TCDF	1.69	0	
12378-PCDF	< 0.14	0.05	< 0.01
23478-PCDF	0.19	0.5	0.09
Summe-PCDF	2.07	0	
123478-HxCDF	0.25	0.1	0.03
123678-HxCDF	0.27	0.1	0.03
123789-HxCDF	0.40	0.1	0.04
234678-HxCDF	< 0.26	0.1	< 0.03
Summe-HxCDF	2.95	0	
1234678-HpCDF	0.92	0.01	0.01
1234789-HpCDF	< 0.33	0.01	< 0.00
Summe-HpCDF	1.24	0	
OCDF	1.43	0.001	0.00
	Summe Tox	[ ng/l TE ]	min. 0.21
	Equivalente TE		max. 0.56

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

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Extraktionsausbeute	2378-TCDD	13012	. 72%
EXII akiioi isausbeule	23/0-1600	13612	. / 4 /0

Bern, 18. Februar 2001 LABOR DR. MEYER AG
Sachbearbeiter:

Dr. G. Meyer

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).

Labor Nr.: Q3782 Probenbezeichnung: HD1 nach Test Scrubber 2

Material: Wasser Projekt Nr. : 344

Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.75	1	< 0.75
Summe-TCDD	< 7.48	0	
1,2,3,7,8-PCDD	< 0.54	0.5	< 0.27
Summe-PCDD	< 5.43	0	
123478-HxCDD	< 0.53	0.1	< 0.05
123678-HxCDD	< 0.49	0.1	< 0.05
123789-HxCDD	< 0.52	0.1	< 0.05
Summe-HxCDD	< 5.15	0	
1234678-HpCDD	< 0.81	0.01	< 0.01
Summe-HpCDD	< 1.62	0	
OCDD	< 8.89	0.001	< 0.01
2378-TCDF	0.13	0.1	0.01
Summe-TCDF	0.83	0	
12378-PCDF	< 0.26	0.05	< 0.01
23478-PCDF	0.16	0.5	0.08
Summe-PCDF	0.81	0	
123478-HxCDF	< 0.39	0.1	< 0.04
123678-HxCDF	< 0.37	0.1	< 0.04
123789-HxCDF	0.45	0.1	0.05
234678-HxCDF	< 0.60	0.1	< 0.06
Summe-HxCDF	1.41	0	
1234678-HpCDF	1.41	0.01	0.01
1234789-HpCDF	< 0.54	0.01	< 0.01
Summe-HpCDF	1.85	0	
OCDF	1.27	0.001	0.00
	Summe Tox	[ ng/l TE ]	min. 0.15
	Equivalente TE		max. 1.50

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

Extraktionsausbeute	2378-TCDD	13012	· 67%
Extraktionsauspeute	23/0-1600	13612	. 0/70

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).

Labor Nr.: Q3783 Probenbezeichnung: HD2 nach Test Scrubber 1

Material: Wasser Projekt Nr. : 344

Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.09	1	< 0.09
Summe-TCDD	< 0.89	0	
1,2,3,7,8-PCDD	0.33	0.5	0.17
Summe-PCDD	2.70	0	
123478-HxCDD	0.47	0.1	0.05
123678-HxCDD	0.93	0.1	0.09
123789-HxCDD	0.65	0.1	0.07
Summe-HxCDD	8.35	0	
1234678-HpCDD	15.24	0.01	0.15
Summe-HpCDD	37.56	0	
OCDD	15.77	0.001	0.02
2378-TCDF	0.41	0.1	0.04
2378-TCDF Summe-TCDF	0.41 3.52	0.1	0.04
12378-PCDF	0.52	0.05	0.03
23478-PCDF	1.57	0.5	0.78
Summe-PCDF	14.74	0.5	0.76
123478-HxCDF	3.45	0.1	0.34
123678-HxCDF	3.10	0.1	0.31
123789-HxCDF	5.03	0.1	0.50
234678-HxCDF	0.24	0.1	0.02
Summe-HxCDF	40.13	0	
1234678-HpCDF	33.17	0.01	0.33
1234789-HpCDF	4.71	0.01	0.05
Summe-HpCDF	62.25	0.01	
OCDF	23.43	0.001	0.02
	Summe Tox	[ ng/l TE ]	min. 2.98
	Equivalente TE		max. 3.06

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

Extraktionsausbeute	2278 TCDD	12012	. 76%
Extraktionsauspeute	23/8-1600	13012	: / 6%

Bern, 18. Februar 2001 LABOR DR. MEYER AG

Dr. G. Meyer

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).

Labor Nr.: Q3784 Probenbezeichnung: HD2 nach Test Scrubber 2

Material: Wasser Projekt Nr. : 344

Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.15	1	< 0.15
Summe-TCDD	< 1.52	0	
1,2,3,7,8-PCDD	< 0.18	0.5	< 0.09
Summe-PCDD	< 1.80	0	
123478-HxCDD	< 0.15	0.1	< 0.02
123678-HxCDD	< 0.14	0.1	< 0.01
123789-HxCDD	< 0.14	0.1	< 0.01
Summe-HxCDD	< 1.45	0	
1234678-HpCDD	< 0.53	0.01	< 0.01
Summe-HpCDD	0.91	0	
OCDD	< 5.13	0.001	< 0.01
2378-TCDF	0.08	0.1	0.01
Summe-TCDF	0.74	0	
12378-PCDF	< 0.13	0.05	< 0.01
23478-PCDF	0.07	0.5	0.04
Summe-PCDF	0.42	0	
123478-HxCDF	< 0.16	0.1	< 0.02
123678-HxCDF	< 0.17	0.1	< 0.02
123789-HxCDF	< 0.19	0.1	< 0.02
234678-HxCDF	< 0.24	0.1	< 0.02
Summe-HxCDF	< 1.91	0	
1234678-HpCDF	0.99	0.01	0.01
1234789-HpCDF	< 0.37	0.01	< 0.00
Summe-HpCDF	1.83	0	
OCDF	0.95	0.001	0.00
	Summe Tox	[ ng/l TE ]	min. 0.05
	Equivalente TE		max. 0.44

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

Extraktionsausbeute	2378-TCDD	13012	· 84%
EXII akiioi isausbeule	23/0-1600	13612	. 04/0

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).

Labor Nr.: Q3785 Probenbezeichnung: HD3 nach Test Scrubber 1

Material: Wasser Projekt Nr. : 344

Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.08	1	< 0.08
Summe-TCDD	< 0.80	0	
1,2,3,7,8-PCDD	0.22	0.5	0.11
Summe-PCDD	1.70	0	
123478-HxCDD	0.29	0.1	0.03
123678-HxCDD	0.57	0.1	0.06
123789-HxCDD	0.34	0.1	0.03
Summe-HxCDD	5.57	0	
1234678-HpCDD	10.46	0.01	0.10
Summe-HpCDD	15.05	0	
OCDD	10.00	0.001	0.01
2070 7005			0.00
2378-TCDF	0.28	0.1	0.03
Summe-TCDF	2.61	0	
12378-PCDF	0.34	0.05	0.02
23478-PCDF	0.94	0.5	0.47
Summe-PCDF	10.25	0	
123478-HxCDF	2.29	0.1	0.23
123678-HxCDF	2.03	0.1	0.20
123789-HxCDF	3.25	0.1	0.33
234678-HxCDF	< 0.16	0.1	< 0.02
Summe-HxCDF	26.74	0	
1234678-HpCDF	21.22	0.01	0.21
1234789-HpCDF	3.19	0.01	0.03
Summe-HpCDF	41.94	0	
OCDF	17.66	0.001	0.02
	Summe Tox	[ ng/l TE ]	min. 1.88
	Equivalente TE		max. 1.98

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

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Extraktionsausbeute	2378-TCDD	13012	· 79%
Extraktionsauspeute	23/0-1600	13612	. / 57/0

Bern, 24. Februar 2001 LABOR DR. MEYER AG
Sachbearbeiter:

Dr. G. Meyer

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).

Labor Nr.: Q3786 Probenbezeichnung: HD3 nach Test Scrubber 2

Material: Wasser Projekt Nr. : 344

Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.19	1	< 0.19
Summe-TCDD	< 1.85	0	
1,2,3,7,8-PCDD	< 0.16	0.5	< 0.08
Summe-PCDD	< 1.58	0	
123478-HxCDD	< 0.18	0.1	< 0.02
123678-HxCDD	< 0.15	0.1	< 0.02
123789-HxCDD	< 0.16	0.1	< 0.02
Summe-HxCDD	< 1.63	0	
1234678-HpCDD	< 0.22	0.01	< 0.00
Summe-HpCDD	3.05	0	
OCDD	< 1.93	0.001	< 0.00
		1	
2378-TCDF	< 0.27	0.1	< 0.03
Summe-TCDF	< 2.70	0	
12378-PCDF	< 0.93	0.05	< 0.05
23478-PCDF	< 0.56	0.5	< 0.28
Summe-PCDF	< 7.44	0	
123478-HxCDF	< 1.03	0.1	< 0.10
123678-HxCDF	< 1.00	0.1	< 0.10
123789-HxCDF	< 1.23	0.1	< 0.12
234678-HxCDF	< 1.67	0.1	< 0.17
Summe-HxCDF	< 12.31	0	
1234678-HpCDF	< 1.67	0.01	< 0.02
1234789-HpCDF	< 4.44	0.01	< 0.04
Summe-HpCDF	< 12.22	0	
OCDF	< 8.33	0.001	< 0.01
	Summe Tox	[ ng/l TE ]	min. 0.00
	Equivalente TE		max. 1.23

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

Extraktionsausbeute	2378-TCDD	13012	· 74%
EXII akiioi isauspeule	23/0-1600	13612	. /4-/0

Sachbearbeiter: .....

Bern, 24. Februar 2001 LABOR DR. MEYER AG

Dr. D. Stadler, dipl. Natw. ETH

Dr. G. Meyer

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).

Labor Nr.: Q3787 Probenbezeichnung: HD4 nach Test Scrubber 1

Material: Wasser Projekt Nr. : 344

Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.15	1	< 0.15
Summe-TCDD	< 1.53	0	
1,2,3,7,8-PCDD	0.17	0.5	0.09
Summe-PCDD	0.72	0	
123478-HxCDD	0.21	0.1	0.02
123678-HxCDD	0.36	0.1	0.04
123789-HxCDD	0.21	0.1	0.02
Summe-HxCDD	3.20	0	
1234678-HpCDD	6.96	0.01	0.07
Summe-HpCDD	16.23	0	
OCDD	7.30	0.001	0.01
2070 TODE	0.47		0.00
2378-TCDF	0.17	0.1	0.02
Summe-TCDF	1.63	0	
12378-PCDF	0.23	0.05	0.01
23478-PCDF	0.57	0.5	0.28
Summe-PCDF	5.99	0	
123478-HxCDF	1.38	0.1	0.14
123678-HxCDF	1.50	0.1	0.15
123789-HxCDF	2.22	0.1	0.22
234678-HxCDF	< 0.27	0.1	< 0.03
Summe-HxCDF	17.47	0	
1234678-HpCDF	12.15	0.01	0.12
1234789-HpCDF	1.73	0.01	0.02
Summe-HpCDF	24.63	0	
OCDF	8.57	0.001	0.01
	Summe Tox	[ ng/l TE ]	min. 1.21
	Equivalente TE		max. 1.39

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

Dr. D. Stadler, dipl. Natw. ETH

Extraktionsausbeute	2378-TCDD	13012	· 78%
Extraktionsauspeute	23/0-1600	13612	. / 0 70

Bern, 24. Februar 2001 LABOR DR. MEYER AG
Sachbearbeiter:

Dr. G. Meyer

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).

Labor Nr.: Q3788 Probenbezeichnung: HD4 nach Test Scrubber 2

Material: Wasser Projekt Nr. : 344

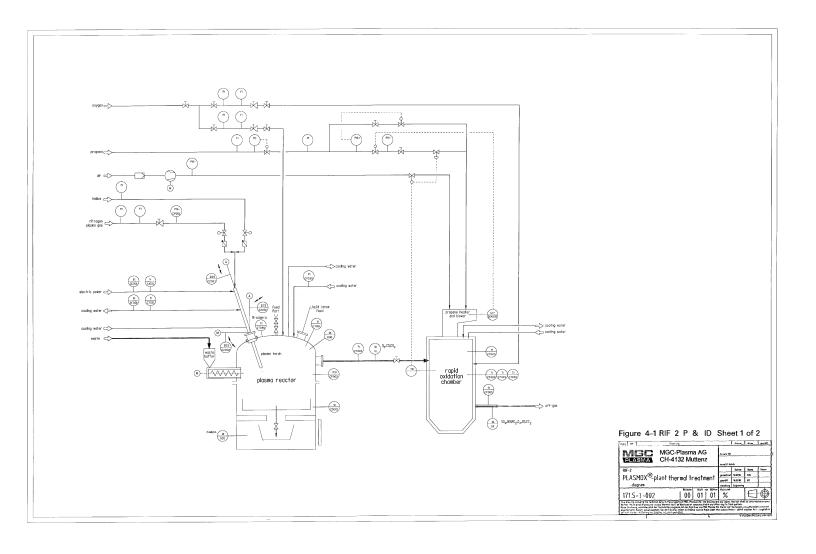
Eingang: 23/ January 2001 Auftraggeber: MGC, Muttenz

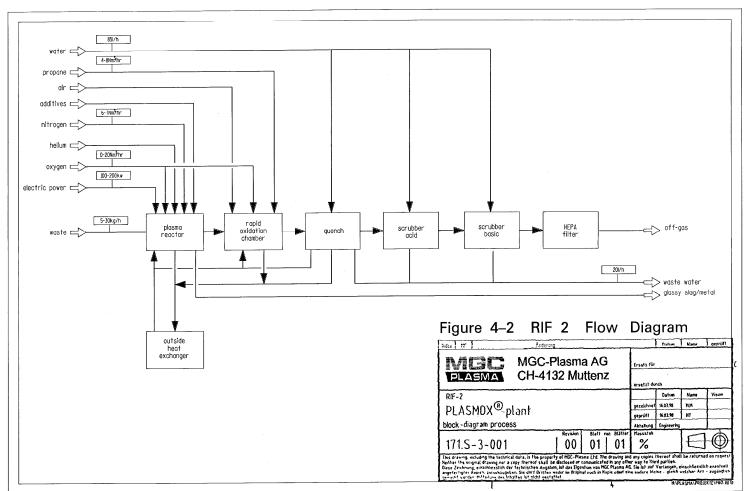
	Gehalt	Tox	Tox. Equivalent
	[ ng/l ]	Faktor *	[ ng/l TE ]
2,3,7,8-TCDD	< 0.19	1	< 0.19
Summe-TCDD	< 1.91	0	
1,2,3,7,8-PCDD	< 0.17	0.5	< 0.09
Summe-PCDD	< 1.74	0	
123478-HxCDD	< 0.16	0.1	< 0.02
123678-HxCDD	< 0.15	0.1	< 0.02
123789-HxCDD	< 0.16	0.1	< 0.02
Summe-HxCDD	< 1.57	0	
1234678-HpCDD	< 0.22	0.01	< 0.00
Summe-HpCDD	< 0.44	0	
OCDD	< 5.38	0.001	< 0.01
2378-TCDF	0.06	0.1	0.01
Summe-TCDF	0.33	0	
12378-PCDF	< 0.13	0.05	< 0.01
23478-PCDF	< 0.08	0.5	< 0.04
Summe-PCDF	< 1.04	0	
123478-HxCDF	< 0.23	0.1	< 0.02
123678-HxCDF	< 0.22	0.1	< 0.02
123789-HxCDF	< 0.28	0.1	< 0.03
234678-HxCDF	< 0.36	0.1	< 0.04
Summe-HxCDF	< 2.71	0	
1234678-HpCDF	< 0.31	0.01	< 0.00
1234789-HpCDF	< 0.61	0.01	< 0.01
Summe-HpCDF	< 1.85	0	
OCDF	< 0.90	0.001	< 0.00
	Summe Tox	[ ng/l TE ]	min. 0.01
	Equivalente TE		max. 0.50

<sup>&</sup>lt; = kleiner als (Zahlenwert = Nachweisgrenze); Nachweisgrenzen sind nur für Einzelisomeren bestimmbar. Die Summen-Nachweisgrenzen sind mittels VDI-Empfehlungen angenäherte Werte.

Extraktionsausbeute	2378-TCDD	13012	. 73%
EXII akiioi isausbeule	23/0-1600	13612	. / 3/0

<sup>\*:</sup> Auswertung mit internationalen Toxizitätsäquivalentsfaktoren ( ITEF ).





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# APPENDIX C

# **Scrubber Efficiency Calculation**

# **APPENDIX C Scrubber Efficiency Estimate**

**Assumptions**: Phosphorus removal efficiency, E, is equal for both scrubbers.

**Data**: Scrubber 1 volume,  $V_1 = 600 L$ 

Scrubber 2 volume,  $V_2 = 400 L$ 

P concentration in Scrubber 1 before test,  $c_{1b} = 0.05 \text{ mg/L}$  P concentration in Scrubber 1 after test,  $c_{1a} = 34.5 \text{ mg/L}$  P concentration in Scrubber 2 before test,  $c_{2b} = 7.00 \text{ mg/L}$  P concentration in Scrubber 2 before test,  $c_{2a} = 25.5 \text{ mg/L}$ 

**Variables:**  $E_1 = \text{Efficiency of Scrubber 1}$ 

 $E_2$  = Efficiency of Scrubber 2

C = amount of phosphorus in ROC exhaust stream

#### **Calculation:**

Quantity of phosphorus collected in Scrubber 1 is equal to amount of phosphorus in ROC exhaust, C, times the efficiency of Scrubber 1,  $E_1$ .

1) 
$$V_1(c_{1a} - c_{1b}) = E_1C$$

Quantity of phosphorus collected in Scrubber 2 is equal to amount of phosphorus exiting Scrubber 1, times the efficiency of Scrubber 2,  $E_2$ .

2) 
$$V_2(c_{2a} - c_{2b}) = E_2(1 - E_1)C$$

Combining Equations (1) and (2):

$$\frac{V_1(c_{1a} - c_{1b})}{V_2(c_{2a} - c_{2b})} = \frac{E_1C}{E_2(1 - E_1)C}$$

From assumptions:

3) 
$$E_1 = E_2 = E$$

Solving for E:

$$[V_1(c_{1a} - c_{1b})] \times (1 - E) = V_2(c_{2a} - c_{2b})$$

$$(1 - E) = V_2(c_{2a} - c_{2b})/V_1(c_{1a} - c_{1b})$$

$$(1 - E) = [(400)(25.5 - 7.0)]/[(600)(34.5 - 0.05)]$$

$$(1 - E) = (7,400)/(20,670) = 0.36$$

$$E = 0.64$$

## APPENDIX D

## **MEA Destruction Calculation**

	Atomic Weights	Formula	Weights			Weight Composit	ion in simulant
				MEA	DMMP	HD	GB
С	12.011	C2H7NO	61.084	244.336		0.83	0.4
Н	1.008	C3H9O3P	124.076		248.152		0.08
0	15.999	O2	31.998	415.974	319.98		
Р	30.974	N2	28.014	-56.028			
N	14.007	CO2	44.009	-352.072	-264.054		
CI	35.453	H2O	18.015	-252.21	-162.135	0.1	0.18
		P2O5	141.943		-141.943		
		HCI	36.461			0.07	
		CO	28.01				

complete combustion eqns

**MEA** 4\*C2H7NO + 13\*O2 = 8\*CO2 + 14\*H20 + 2\*N2

2\*C3H9O3P + 10\*O2 = 6\*CO2 + 9\*H20 +P2O5

assume Normal temperature is 68F

1 gm-mole 22.414 liters (STP)

**Correction Factor** 

V ntp = V stp \* 1.073171

Oxygen Flow to Reactor/hr		N2 PURGE to reactor	
GB-2	6385.22 gm/hr	6.7 NM3/HR	gm/hr
HD-2	7449.424 am/hr	6.7 NM3/HR	am/hr

Simulant Flow density 1.015 kg/L 1.003 kg/L GB-2 HD-2 12.3 Liters/hr 6 Liters/hr

Mass Flo	w (gm/hr)			
	MEA	DMMP	Water	HCI
GB-2 HD-2	4993.8	998.76	2247.21	
HD-2	4994.94		601.8	421.26

					FRACTION			
Can Cor	mplete combustion occur in reactor? Sufficient oxygen		С	H2	0	Z	T	F
		HD	163.5433		261.1889	1.597063	0.402937	0.597063
GB-2	-3404.41 no, partial combustion	GB		322.3586	317.3482	0.984457	0.984457	0.015543
HD-2	-1054.3 no. partial combustion							

ASSUME MAXIMUM VOL P	ARTIAL COMBUSTION	- ONLY CO, SOME H20	, H2 AND N2 FOR HD	SIMULANT	
FIRST WATER THEN SPLIT	CO & CO2				
4994.94	7449.424	5155.908	1845.795	4297.286	1145.376
C2H7NO +	O2 =	H20 +	CO +	CO2 +	N2
61.084	31.998	18.015	28.01	44.009	28.014

ASSUME MAXIMUM VOL PA	ARTIAL COMBUSTION	- ONLY P4,	CO, SOME	CO2, H20 AN	ID N2 FOR	R GB SIMUL	.ANT					
FIRST CO, THEN SPLIT H2	& H20											
4993.8	998.76	6385	.22	5717.0	28	5256.2	209	10.10	095	249.3	278	1145.114
C2H7NO +	C3H9O3P +	O2	=	H20	+	co	+	H2	+	P4	+	N2
61.084	124.076	31.9	998	18.0	115	28	.01	2.	016	123.	896	28.014

O2 BAL REACTANTS H2O CO CO2 4578.927 1054.297 3124.464 8757.688

REACTANTS CO H20 H2 3002.288 5077.254 8079.542

Destruction Efficience	су	0.9999991	0.9999992
MEA EFFLUENT FRO	OM REACTOR	4.5903317 MG	3.9166176 M
TOTAL GAS FLOW	NM3/HR	22.951658	19.583088
N2	NM3/HR	0.9832453	0.9834697
P4	NM3/HR	0	0
CO2	NM3/HR	0	2.3487724
CO	NM3/HR	4.5138563	1.5851065
H2	NM3/HR	0.1205202	0
H20	NM3/HR	7.6335094	6.8842881
REACTANTS			
HCI	NM3/HR	0	0.2779136
WATER	NM3/HR	3.0005273	0.8035374
N2 PURGE	NM3/HR	6.7	6.7
		GB	HD
GASEOUS FLOW			